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Organic Analyses in Water Quality Control Programs; Instructor's Guide for Conducting the Course

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This manual is designed for use by instructors who will have to teach others how to perform analyses for selected organic pollutants. It is to be used in conjunction with the training manual bearing the same title (EPA-430/1-80-011). It includes topics related to the presentation of training courses in which the analyses would be taught. These topics include: the course description, personnel needed to conduct the training, the course agenda, a milestones list for planning and preparing the course, instructional resources available to support the instruction and a list of equipment and supply requirements. Also included are Instructional Package Worksheets (lesson plans) giving details relative to presenting instruction regarding each of the analyses covered in the assoicated student reference manual.

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Instructor's Guide

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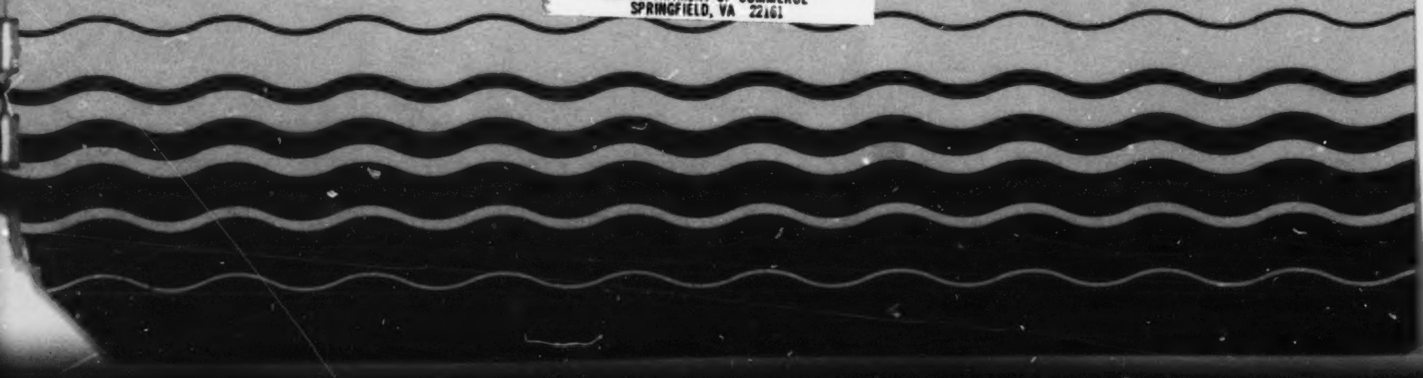

INSTRUCTOR'S GUIDE

for

CONDUCTING THE COURSE

ORGANIC ANALYSES

IN

WATER QUALITY CONTROL PROGRAMS

National Training and Operational Technology Center
Office of Water Program Operations
U. S. Environmental Protection Agency

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Preface

Introduction

This is one of several courses offered by the National Training and Operational Technology Center (NTOTC) which have been prepared in packaged form for use by the States in their training programs, and by other organizations having a need to present this type of training. Each course package consists of:

1. An instructor's guide containing material related to course planning and conduct,
2. a training manual for use by the participants, containing the course subject matter, and
3. supportive visual and audiovisual training aids used by NTOTC in presenting the course. These are available on loan to those offering the course. The content of each slide is reproduced in this manual.

This guide reflects the manner in which the course has been offered by NTOTC. It is intended to assist the organization offering the training, and should not be considered as an inflexible method of presenting the course. Some may want to follow the format exactly as presented--others may not. In either case, this guide should prove helpful in reducing the amount of orignial developmental work required, and in suggesting methods and approaches when modifications of the course plan presented herein are being considered.

Organization of the Guide

This instructor's guide consists of two major parts. Part I contains information required for course planning and management. Part II consists of a series of Instructional Package Worksheets (IPW's) which set forth learning objectives and the instructional approach used by NTOTC for each topic included in the training manual.

Analytical Methodology for Regulatory Programs

It is essential that analytical procedures taught in the course conform to those prescribed for use in USEPA regulatory programs. These procedures are identified in regulations which appear first in the Federal Register, and which are later codified in Title 40 of the annual edition of the Code of Federal Regulations.

For the National Pollutant Discharge Elimination System (NPDES), Part 136 of Title 40, "Guidelines Establishing Test Procedures for the Analysis of Pollutants", specifies methods to be used for the measurement of contaminants in wastewater.

For drinking water, Part 141 of Title 40, "National Interim Primary Drinking Water Regulations" (NIPDWR), specifies methods to be used in determining the level of contaminants in finished water.

The Instructional Package Worksheet on Compliance Monitoring in Part II of this guide provides details concerning EPA regulations for these programs.

For additional information concerning this course, or other packaged courses, contact:

Director, NTOTC
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

PART I COURSE PLANNING AND MANAGEMENT

This section of the manual is concerned with the administrative aspects of planning, preparing, and conducing the course.

A. COURSE DESCRIPTION

This description was prepared for course presentation at the NTOTC and may be useful to others in course-related publicity.

ORGANIC ANALYSES IN WATER QUALITY CONTROL PROGRAMS

5 days

This course is for chemists and technicians with little or no experience in organic analyses commonly required for NPDES and NIPDWR regulations and for other water quality programs. Applicants should have a fundamental knowledge of organic chemistry and quantitative analyses, and should have basic laboratory skills including the use of volumetric glassware and titration assemblies. They should be actively engaged in a water quality control program.

After successfully completing the course, the student will know how to perform analyses for selected organic pollutants listed as approved in the Federal Register.

The training is a five-day course which includes classroom instruction, student performance of laboratory procedures, and discussion of each laboratory assignment and reported results.

Course topics include NPDES methodology, laboratory safety, dissolved oxygen (Winkler), five-day biochemical oxygen demand, total organic carbon, oil and grease (separatory funnel extraction), surfactants (MBAS), total Kjeldahl and organic nitrogen (Nesslerization), phenol (4-AAP), polychlorinated biphenyls, pesticides, and control of analytical performance. Emphasis is on laboratory operations.

B. PERSONNEL

The remaining sections in Part I of this manual indicate the personnel associated with this training course when it is presented at the National Training and Operational Technology Center (NTOTC). Their course-related activities are listed below.

1. Training Supervisor -
   Has overall responsibility for the NTOTC training program.

2. Training Coordinator -
   Is responsible for all elements involved in planning and conducting a specific course.

3. Course Secretary -
   Performs all course-related clerical duties.

4. Instructor -
   For assigned topics, is responsible for planning instructional approach, developing instructional materials, and delivering the instruction. During laboratory sessions, another instructor may be designated to assist the (primary) instructor so that participants can be provided as much individualized attention as possible.

5. Laboratory Assistant -
   Assists in the prepartion of laboratory reagents; assembles equipment; is available as required during laboratory exercises.

C. SUMMARY PLAN FOR THE COURSE AND COURSE SCHEDULE

A convenient format to use in the early stages of devising a course plan is a day-to-day assignment of time blocks based on estimates by instructors of the training time required for each parameter. (An example is on the next page.) Using available time as a first criterion will allow a variety of possible sequences. One such sequence, which has been successfully used by NTOTC to conduct this course in the past, begins on page 2-3. Examples of other considerations are:

1. If some equipment must be used in more than one test, schedule another topic between the two tests to allow time for the required clean-up.
2. Schedule the topics so each instructor alternates between prime and assistant responsibilities to allow time for preparations which must be done right before training sessions.
3. If one procedure requires skills taught in another procedure, order the presentations accordingly.

SUMMARY PLAN FOR THE COURSE

SUMMARY PLAN FOR THE COURSE

MONDAY		TUESDAY		WEDNESDAY		THURSDAY		FRIDAY	
Activity	Time Hours	Activity	Time Hours	Activity	Time Hours	Activity	Time Hours	Activity	Time Hours
Registration	1/4	Carbon Analyses	3/4	Lab - COD	1	Lab - Group A, Surfactants	2	Poly. Biphenyls	1/2
Welcome & Course Objectives	1/2	Nitrogen Cycle	1/2	Demo - TOC	1/2	Lab - Group B, Chlor. Hydrocarbons & GC	-	Demo-Poly. Biphen	1½
Sample Handling	1/2	Kjeldahl Nitrogen	1/2	Surfactants	3/4	Oil & Grease	3/4	Analytical Per.	3/4
Compliance Meth.	1	Lab-Kjeldahl Nit.	1/4					Post-Results	1
Lab Safety	1/2	Ammonia Nitrogen	1/2					Analytical Per.	1/2
		Spec 20 & Cal. Graphs	3/4					Lab - BOD	1/2
Lunch	1	Lunch	1	Lunch	1	Lunch	1	Evaluation & Closing	1/4
DO - Winkler	3/4	Lab-Kjeld. Nit.	1¼	Gas Chromatog.	3/4	Phenol	3/4		
DO - Meter	3/4	COD	3/4	Lab - Group A, Chlor. Hydrocarbons & GC	2	Lab - Group A, Oil & Grease	2		
BOD	3/4	Lab - COD	1/4	Lab - Group B, Surfactants	-	Lab - Group B, Phenol	-		
Lab - BOD	1 3/4					Lab-Gp A -Phenol	2		
						Lab - Group B, Oil & Grease	-		
TOTAL	9*	TOTAL	8¼*	TOTAL	7½*	TOTAL	9½*	TOTAL	6*

*Includes quizzes, discussions, laboratory briefings, and breaks not shown above.

ORGANIC ANALYSES IN WATER
QUALITY CONTROL PROGRAMS (100.5)

(Location)
(Date)

AGENDA

Course Coordinator:

Laboratory Assistant:

DAY & TIME	SUBJECT	OUTLINE	INSTRUCTOR*
Monday			
8:30 - 8:45	Registration		Course Secretary
8:45 - 8:50	Welcome		Course Coordinator
8:50 - 9:20	Course Objectives		Course Coordinator
9:20 - 9:50	Sample Handling - Field through Laboratory	2	Chemist 3
9:50 - 10:10	Break		
10:10 - 11:10	Compliance Methodology	3	Chemist 3
11:10 - 11:30	Quiz 1. Sample Handling - Field through Laboratory 2. Compliance Methodology		Chemist 3
11:30 - 12:00	Laboratory Safety Practices	1	Chemist 2
12:00 - 1:00	Lunch		
1:00 - 1:40	Dissolved Oxygen (DO) - Winkler	8, 9, 10	Chemist 1
1:40 - 2:20	Dissolved Oxygen (DO) - Meter	11	Chemist 1
2:20 - 3:05	Biochemical Oxygen Demand (BOD)	12, 13	Chemist 1
3:05 - 3:20	Break		
3:20 - 3:35	Laboratory Briefing		Chemist 1
3:35 - 5:25	Laboratory 1. Set up BOD 2. Initial DO Titration	10, 13	Chemist 1 (Chemist 3)
Tuesday			
8:30 - 8:45	Discussion 1. DO 2. BOD		Chemist 1
8:45 - 9:30	Carbon Analyses	17	Chemist 1
9:30 - 9:45	Break		
9:45 - 10:15	Nitrogen Cycle	15	Chemist 2
10:15 - 10:45	Total Kjeldahl Nitrogen	14	Chemist 2
10:45 - 11:05	Laboratory - Kjeldahl Digestion	16	Chemist 2 (Chemist 1)

DAY & TIME	SUBJECT	OUTLINE	INSTRUCTOR*
Tuesday (Cont'd.)			
11:05 - 11:25	Ammonia Nitrogen	15	Chemist 2
11:25 - 12:10	Use of a Spectronic 20 and Preparation of Calibration Graphs	7	Chemist 1
12:10 - 1:10	Lunch		
1:10 - 1:40	Laboratory Briefing		Chemist 2
1:40 - 2:50	Laboratory 1. Kjeldahl Distillation 2. Ammonia Calibration Curve	16	Chemist 2 (Chemist 3)
2:50 - 3:05	Break		
3:05 - 3:25	Discussion and Quiz - All Nitrogen Topics		Chemist 2
3:25 - 4:10	Chemical Oxygen Demand COD)	18	Chemist 3
4:10 - 4:25	Laboratory Briefing		Chemist 3
4:25 - 4:45	Laboratory - COD	19	Chemist 3 (Chemist 2)
Wednesday			
8:30 - 8:40	Laboratory Briefing		Chemist 3
8:40 - 9:45	Laboratory 1. Standardize Ferrous Ammonium Sulfate 2. Titrate COD Sample 3. Calculation of Results	19	Chemist 3 (Chemist 2)
9:45 - 10:00	Break		
10:00 - 10:15	Discussion and Quiz - Chemical Oxygen Demand		Chemist 3
10:15 - 10:50	Laboratory Demonstration - Total Organic Carbon		Chemist 1
10:50 - 11:00	Discussion and Quiz - Total Organic Carbon		Chemist 1
11:00 - 11:45	Surfactants	20	Chemist 1
11:45 - 12:45	Lunch		
12:45 - 1:30	Introduction to Gas Chromatography	26, 27	Chemist 2
1:30 - 2:00	Laboratory Briefing		Chemist 1
2:00 - 4:00	Laboratory - Group A 1. Extraction of Chlorinated Hydrocarbons 2. Gas Chromatography	30	Chemist 1 (Chemist 2)
2:00 - 4:00	Laboratory - Group B Surfactants	21	Chemist 3

DAY & TIME	SUBJECT	OUTLINE	INSTRUCTOR*
Thursday			
8:30 - 8:35	Laboratory Briefing		Chemist 1
8:35 - 10:35	Laboratory - Group A Surfactants	21	Chemist 3
8:35 - 10:35	Laboratory - Group B 1. Extraction of Chlorinated Hydrocarbons 2. Gas Chromatography	30	Chemist 1 (Chemist 2)
10:35 - 10:50	Break		
10:50 - 11:05	Discussion and Quiz 1. Surfactants 2. Extraction of Chlorinated Hydrocarbons/ Gas Chromatography		Chemist 1 (Chemist 2)
11:05 - 11:50	Oil and Grease	22	Chemist 1
11:50 - 12:50	Lunch		
12:50 - 1:35	Phenol	24	Chemist 3
1:35 - 2:05	Laboratory Briefing		Chemist 1
2:05 - 4:10	Laboratory - Group A Oil and Grease	23	Chemist 3
2:05 - 4:10	Laboratory - Group B Phenol	25	Chemist 1
4:10 - 4:15	Laboratory Briefing		Chemist 1
4:15 - 6:20	Laboratory - Group A Phenol	25	Chemist 1
4:15 - 6:20	Laboratory - Group B Oil and Grease	23	Chemist 3
Friday			
8:30 - 8:50	Discussion and Quiz 1. Oil and Grease 2. Phenol		Chemist 1 (Chemist 3)
8:50 - 9:20	Polychlorinated Biphenyls	28	Chemist 2
9:20 - 9:35	Break		
9:35 - 11:05	Laboratory Demonstration - Polychlorinated Biphenyls	29	Chemist 2
11:05 - 11:50	Control of Analytical Performance	6	Chemist 3
11:50 - 12:50	Lunch and Posting of Analytical Results		
12:50 - 1:15	Control of Analytical Performance (Cont'd.)	6	Chemist 3

DAY & TIME	SUBJECT	OUTLINE	INSTRUCTOR*
Friday (Cont'd.)			
1:15 - 1:25	Discussion and Quiz - Control of Analytical Performance		Chemist 3
1:25 - 1:30	Laboratory Briefing		Chemist 1
1:30 - 1:55	Laboratory 1. Final DO Titration 2. Calculation of BOD_4		Chemist 1
1:55 - 2:10	Discussion and Quiz - BOD		Chemist 1
2:10 - 2:25	Course Evaluation and Closing		

*For the actual course presentation, insert the name of the primary instructor opposite the title of his/her assignment. Assistant instructors are designated by (   ).

D. MILESTONES IN COURSE PLANNING AND PREPARATION

The following pages list major areas of course responsibilities in a chronological order to facilitate orderly and timely accomplishment. The table also serves as an example for assignment of these responsibilities to various staff members. It has been successfully used by NTOTC to conduct this course in the past.

The table headings are job titles associated with the listed tasks. A suggested staff is cited, including a laboratory assistant. It is recognized, however, that staff is often limited and one individual may serve in several of the defined roles. Having this summary according to an ideal situation should facilitate an equitable division of the required tasks among fewer persons.

Before using the milestones table, decisions must be made about the course content. It may be desirable to teach the approved test procedures for parameters that are not included in this package, but that are required locally to meet regulatory requirements. In that case, the table must be changed. Delete items identified for topics you omit and add the items needed for the topics you want to add.

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
5 TO 6 MONTHS BEFORE COURSE							
Determination of the need and decision to have course.	x						
Designation of Course Director and Course Secretary.	x						
Review responsibilities.		x					
Review responsibilities.			x				
Commit classroom and laboratory facilities.	x	x					
Develop and release Course Announcement including location, date, general statement of course content and training objectives.		x	x				
Prepare all forms and information sheets related to student registration procedures.		x	x				
Decide on staff members.	x	x					
4 TO 5 MONTHS BEFORE COURSE							
Receive, review, act upon Course Applications, continuing until course begins.		x	x				
Maintain records on disposition of each application, continuing through course.			x				
Inventory Instructor's Guides, Order needs.		x	x				

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
3 MONTHS BEFORE COURSE							
Commit all staff members who will participate in Course.	x	x		x	x	x	x
Develop Milestone Checklist for Course.		x	x				
Distribute copies to staff of Milestones, Instructor's Guide, Student Text and any other pertinent training resources.	x	x	x				
Review responsibilities.				x	x	x	x
Assign topics to Primary (P) and Assistant (A) Instructors:	x	x					
Sample Handling						P	
Compliance Methodology						P	
Lab Safety Practices					P		
Diss. Ox & Bio. Ox Demand				P		A	
Carbon Analyses				P			
Nitrogen Topics					P	A	
Spec 20 & Cal. Graphs				P			
Chemical Oxygen Demand					A	P	
Surfactants				P		A	
Intro to Gas Chromatography				A	P		
Oil & Grease				P			
Phenol						P	
Polychlorinated Biphenyls					P		
Control of Analyt. Perform						P	
Develop summary plan for course.		x	x	x	x	x	x
Inventory chemicals and laboratory equipment/supplies. List and commit lending sources. Order rest of needs.		x	x	x	x	x	x
Inventory classroom equipment/supplies. List and commit lending sources. Order rest of needs.		x	x				

(Continued)

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
Inventory student reference texts. Order needs.		x	x				
2 MONTHS BEFORE COURSE							
Finalize Course Schedule (Agenda).		x	x	x	x	x	x
Request laboratory/classroom needs from lending sources.		x	x	x	x	x	x
Request training aids from lending sources.		x	x	x	x	x	
6 WEEKS BEFORE COURSE							
Check out operation of all major pieces of equipment.				x	x	x	x
Primary and Assistant Instructors go through laboratory procedures in student reference texts, using IPWs to standardize instructions for students.				x	x	x	
1 MONTH BEFORE COURSE							
Summary (to date) to staff of registered students, continuing to course beginning.		x	x				
Check on progress of staff preparations for instruction, continuing through course.		x		x	x	x	
Prepare all administrative forms and materials needed for course presentation.		x	x				
Plan and rehearse classroom presentations using all required training aids. Finalize.				x	x	x	

(Continued)

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant	
Obtain any duplicated instructional materials (data sheets, etc.).			x	x	x	x	x	
Review summary of laboratory equipment and supply needs for expected number of students doing the selected procedures.		x					x	
Clean all glassware required by students. Note any special requirements.							x	
Reserve all specially-cleaned glassware.							x	
Assemble other student equipment and supplies.							x	
2 WEEKS BEFORE COURSE								
Arrange for security of classroom and laboratory.		x	x					
Make reagents required by students EXCEPT those with specified, limited stability.				x	x	x	x	
Make final arrangements to obtain required samples. Plan for any "synthetics."				x	x	x	x	
Determine range of concentration of desired constituent(s) in samples for the course.				x	x	x		
Arrange for disposal of special test wastes.				x	x	x		
(Continued)								

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
Give Laboratory Assistant final list of equipment and supplies to be at each laboratory position. Discuss arrangement of shared equipment.				x	x	x	
1 WEEK BEFORE COURSE							
Inform building food service of number of expected students and course lunch times (as appropriate).			x				
3 DAYS BEFORE COURSE							
Finalize seating arrangement for classroom.		x					
Assemble course materials in classroom (student texts, administrative materials, etc.). Distribute as appropriate.		x	x				
Ready classroom instructional aids (boards, erasers, etc.)		x	x				x
Check out all classroom equipment (electrical systems, PA, projection equipment) and obtain back-up accessories (bulbs, etc.).		x					
COURSE OPENING							
Conduct opening exercises.		x					
Participate in course opening.	x		x	x	x	x	x
Complete any required student records, including roster.		x	x				
(Continued)							

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
Prepare course certificates if needed at end of week.		x	x				
EVERY DAY OF COURSE							
Maintain general supervision of course.		x					
Prepare unstable reagents and/or samples on day of test.				x	x	x	x
Obtain samples for each test on day of test.				x	x	x	x
When assistant instructor, make any student evaluation records requested by the primary instructor.				x	x	x	
When primary instructor, compile evaluation record for each student.				x	x	x	
Keep any general records (e.g. attendance) as required to document successful course completion.		x		x	x	x	
Oversee disposal of special test wastes.				x	x		x

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
SECOND-LAST DAY OF COURSE							
Distribute course critique sheet to students.		x					
LAST DAY OF COURSE							
Check and sign course certificates if applicable		x					
Collect students' critique sheets.		x					
Conduct closing exercises.		x					
Participate in course closing.	x		x	x	x	x	x
Clean up classroom and laboratory.		x	x	x	x	x	x
WITHIN TWO WEEKS OF COURSE PRESENTATION							
Return or replace any borrowed classroom equipment/ supplies.		x	x				
Return or replace any borrowed laboratory equipment/ supplies.				x	x	x	x
Return or replace any borrowed training aids.		x	x	x	x	x	
Order repairs or replacements of own equipment used in course (if necessary).		x	x	x	x	x	
Complete and file evaluation records on all students in predetermined area.			x	x	x	x	
See that all students have been appropriately notified of their degree of success in completing the course.		x	x				

	Training Supervisor	Course Coordinator	Course Secretary	Instructor #1	Instructor #2	Instructor #3	Lab Assistant
Participate in staff session on evaluation of course and recommendations for future offerings.	x	x	x	x	x	x	x
Prepare course summary/ evaluation report.		x					
Complete and file entire course records in mutually determined area.		x	x				

E. INSTRUCTIONAL RESOURCES

Most training institutions will make the fullest possible use of available instructional resources. The purpose of this section is to describe the nature, sources and availability of instructional resources suggested for use with this course.

1. Student reference text and Instructor's Guide for the course, "Organic Analyses in Water Quality Control Programs." Ordering information may be obtained from:

   U.S. Environmental Protection Agency
   National Training and Operational Technology Center
   Cincinnati, Ohio 45268
   (513) 684-7501

2. Slides and Slide/Tape Units (See IPW Sections VIII B and XI for a description).

   a) What is available, according to topic:

      (1) Sample Handling - Field Through Laboratory

         (a) X-17, Sample Handling, 8 slides

      (2) Laboratory Safety Practices

         (a) X-27, Laboratory Safety Practices, 40 slides

      (3) Dissolved Oxygen (DO) - Winkler, Dissolved Oxygen (DO) - Meter, Biochemical Oxygen Demand (BOD)

         (a) XT-31, Dissolved Oxygen - General Considerations, 7 slides

         (b) XT-29, Dissolved Oxygen Determination, slide/tape unit, 72 slides, 15 minute tape

         (c) X-32, YSI DO Meter, 29 slides

         (d) XT-54, The Determination of Biochemical Oxygen Demand, slide/tape unit, 71 slides, 13 minute tape

      (4) Carbon Analyses

         (a) XT-59, Determination of Total Organic Carbon, slide/tape unit, 62 slides, 13 minute tape

      (5) Nitrogen Cycle

         (a) X-35, Kjeldahl Nitrogen, 10 slides

(6) Total Kjeldahl Nitrogen, 10 slides

  (a) X-35, Kjeldahl Nitrogen, 10 slides

(7) Ammonia Nitrogen

  (a) X-35, Kjeldahl Nitrogen, 10 slides

  (b) XT-67, Determination of Ammonia Nitrogen, slide/tape unit, 45 slides, 8 minute tape

(8) Use of a Spectronic 20 and Preparation of Calibration Graphs

  (a) X-8, Use of a Spectrophotometer, 16 slides

  (b) X-9, Calibration Graphs, 6 slides

  (c) XT-51, Use of the Spectronic 20 Spectrophotmeter, slide/tape unit, 35 slides, 10 minute tape

(9) Chemical Oxygen Demand

  (a) XT-27, Chemical Oxygen Demand, slide/tape unit, 60 slides, 17 minute tape

(10) Surfactants

  (a) X-34, Surfactants, 20 slides

(11) Introduction to Gas Chromatography

  (a) X-36, Gas Chromatography, 5 slides

(12) Oil and Grease

  (a) XT-56, Determination of Grease and Oil, slide/tape unit, 54 slides, 15 minute tape

(13) Phenol

  (a) X-37, Phenols, 4 slides

  (b) XT-28, Determination of Phenolics, slide/tape unit, 65 slides, 17 minute tape

(14) Control of Analytical Performance

  (a) X-24, Precision, 20 slides

  (b) X-25, Accuracy, 14 slides

  (c) X-26, Quality Assurance, 5 slides

All units described in 2. above are available on scheduled loan from NTOTC to institutions conducting this course. Requests should contain the information items on the "Request for Loan" form at the end of this section. Send requests to the National Training and Operational Technology Center at the address on page 4-1.

It is urged that materials desired from NTOTC for a specific course offering be requested in a single, consolidated communication. This will give greatest assurance of a well-coordinated response. Because these requests ordinarily will cover a number of different items, telephonic requests should not be made.

Requests should be timely. To assure effective delivery in time for use in the course, requests should be received at NTOTC at least 45 days prior to the course date. NTOTC will, in turn, make every effort to assure that the requested materials are delivered to the requesting institution several days prior to the start of the course in which they are to be used. This will permit review and practice by the instructional staff for the most effective use of such resources.

It is expected that all borrowed resources be returned to NTOTC within two weeks after completion of the course in which they are used.

With returned borrowed training resources, it is requested that the user provide NTOTC with an evaluation of the training resource(s) used. In this manner, the experience of users can be a factor in continuous improvements and responses to problems in using the resources. All reports on the use of such resources should include the number of students with whom the material was used.

3. Supportive References:

a) Manual: EPA-EMSL, "Methods for Chemical Analysis of Water and Wastes". This is the reference source for many of the methods presented in this course. Address requests for a copy to:

U.S. Environmental Protection Agency
Environmental Monitoring and Support Laboratory (EMSL)
Cincinnati, Ohio 45268

b) "Standard Methods for the Examination of Water and Wastewater" (14th edition), APHA, AWWA, WPCF. Available from Publication Office, American Public Health Association, Inc., 1015 18th Street, N.W., Washington, DC 20036.

c) "Annual Book of Standards" Part 31, "Water", 1975. Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

d) Catalog: "Water Quality Control Instructional Materials." This includes visual instructional units developed by NTOTC. Although not developed specifically for this course, several of the units are on Course topics and might be useful supplemetnary material. Address requests for a copy to NTOTC at the address given in E.1. on page 4-1.

   Note: Items 3.a)., b)., and c). are those currently cited by the Federal Registers for National Pollutant Discharge Elimination System (NPDES) and National Interim Primary Drinking Water Regulations (NIPDWR) (copies are in the Instructional Package Worksheet on Compliance Methodology). Contact your U.S. EPA Regional Quality Assurance Coordinator for pertinent Federal Registers issued after 8/80. (Call U.S. EPA, EMSL, at 513/684-7301, for the name and phone number of your Coordinator).

4. Instructional Resources Already in Possession of the Training Institution

   a) Many training organizations prefer to develop, or have developed, their own texts and audiovisual training resources.

REQUEST FOR LOAN
AUDIOVISUAL INSTRUCTIONAL UNIT

Title and Catalog No. ______________________________

____

Intended Use: ______________________________

____

Preferred Date of Use: ______________________________

Alternate Date: ______________________________

BORROWER'S NAME ______________________________

Title ______________________________

Organization ______________________________

Address ______________________________

____
(Zip)

Phone Number (include
Area Code): ______________________________

There is no charge for use of the Audiovisual Instructional Units. However, the *BORROWER* assumes financial responsibility for the value of all loaned equipment and instructional materials.

Unless special arrangements are made with the loaning office, units should be returned within two weeks. Return the unit by *REGISTERED, CERTIFIED* or *INSURED MAIL IMMEDIATELY* after use.

EPA-171 (Cin)
(8-74)

F. LABORATORY EQUIPMENT AND SUPPLY REQUIREMENTS

The consolidated list in this section is for overall planning purposes. It was compiled from the Instructional Package Worksheet (IPW) sections (part II of this manual), IX, "IPW Equipment and Supply Requirements" and X, "IPW Reagent Requirements." Sections IX and X can also be used on a day-to-day basis during the course to prepare for the laboratory exercises.

The first column of this consolidated list contains a description of the item as found in commercial catalogs.

The second column identifies the tests utilizing the equipment. At the beginning of the table, there is a key to the abbreviations used in this column.

The third column lists the minimum quantity per laboratory station (no more than two persons) required for each test. In many cases, multiple use of the item is required to carry out the procedure. The quantities are based on the assignment stated in section X of the related IPW. For convenience, a list of the assignments for each topic can be found as part of the key for abbreviations at the beginning of the table. If an Instructor chooses a different assignment for a topic, some quantities must be changed accordingly. As noted, numbers represent minimum quantities. It is strongly recommended that Instructors provide surplus equipment and additional supplies ready for use in case of need. Many Instructors plan for a margin of at least 10% of extra supplies to provide for student errors, planning miscalculations, or other unforeseen events.

The fourth and final column contains remarks that may be useful when deciding on class needs or when ordering equipment.

Before using the list, decisions must be made about the course content. It may be desirable to teach the approved test procedures for parameters that are not included in this package, but that are required locally to meet regulatory requirements. In that case, the list must be changed. Delete items identified for topics you omit and add the items needed for the topics you want to add.

This list can be of great value in pre-course planning, to determine the availability of needed equipment and supplies, and to take action to provide needed resources. Further, this list can be of vital importance when planning for courses to be conducted in field locations. Copies of the list in the hands of the Course Coordinator and a representative of the host organization can be used to determine which will provide needed resources on an item-by-item basis. When the responsibility is assigned/accepted, this can be annotated in the "remarks" column on the copy in the hands of the Course Coordinator and the copy of the representative of the host organization. Each can then use the annotated equipment and supply list as a checklist for carrying out his own agreed - upon responsibilities in preparing for the course.

LABORATORY EQUIPMENT AND SUPPLY REQUIREMENTS

Following is the key for abbreviations used for course topics. Quantities per test are based on the IPW assignments as noted.

ABBREVIATIONS FOR TESTS		IPW ASSIGNMENTS
DO (W)	Dissolved Oxygen-Winkler	1 sample
DO (M)	Dissolved Oxygen-Meter	1 sample
BOD	Biochemical Oxygen Demand	1 sample
CA	Carbon Analyses	1 sample
KD	Kjeldahl Digestion	1 sample
KD & ACC	Kjeldahl Distillation & Ammonia Calibration Curve	1 sample + 1 standard (0 or other)
CG	Calibration Graphs	As noted in the individual IPW's
SP	Spectrophotometer	As noted in the individual IPW's
COD	Chemical Oxygen Demand	1 sample or blank
SUR	Surfactants	1 sample + 3 standards (0 + 2 others)
GC & ECH	Gas Chromatography & Extraction of Chlorinated Hydrocarbons	1 sample + 3 standards
OG	Oil & Grease	1 sample
PHEN	Phenol	1 sample + 3 standards (0 + 2 others)
PCB	Polychlorinated Biphenyls	1 sample + 1 standard

Regarding the Quantity per Station column on the following pages, no more than two persons should work at a station.

(Compiled from section IX of the IPW's)

DESCRIPTION	TEST	QUANTITY PER STATION	REMARKS
ADAPTER, offset, 24/40 ground glass joints, with small stopper to plug the open hole. (SGA JA-4950 or equivalent)	PHEN	1	Part of distillation apparatus.
APRON (or coat), laboratory	All	2	
BALANCE, analytical	OG	See Remarks	1 per 4 stations.
BALANCE, trip, 0.01 g sensitivity	DO (W), BOD	See Remarks	1 per 4 stations.
BEAKER, 100 ml	GC & ECH	1	
BEAKER, 150-250 ml	COD	2	
BEAKER, 1 liter	PHEN	1	
BOTTLES, BOD, 300 ml ± 3 ml	BOD	2	
BOTTLES, wash, plastic squeeze type	All	1	
BURET, 25 ml	DO (W), BOD	1	Teflon stopcock.
BURET, 50 ml	COD	1	Teflon stopcock.
BURNER, Fisher (or equivalent)	PHEN	1	Part of disitllation apparatus.
CARBON ANALYZER	CA	See Remarks	1 per class. Capable of measuring total organic carbon.
CELL, spectrophotometric, 1 cm	KD & ACC SUR PHEN	1 1 1	For use with Spectronic 20, or equivalent. For use with Spectronic 20, or equivalent. For use with Spectronic 20, or equivalent.
CLAMP,	GC & ECH	1	To support the Kuderna-Danish apparatus.
CLAMP, buret	DO (W), BOD COD	1 1	
CLAMP, 3-finger	PHEN	2	To support the phenol distillation apparatus

DESCRIPTION	TEST	QUANTITY PER STATION	REMARKS
CONDENSER, Liebig (or equivalent), 300 mm jacket (with tubing), 24/40 ground glass neck.	COD	1	Part of reflux apparatus.
CONDENSER, 40 cm (with tubing), 24/40 ground glass joints (SGA JC-9375 or equivalent).	PHEN	1	Part of distillation apparatus.
CYLINDER, graduated, 1 liter	DO (W), DO (M), COD OG	1 1	
CYLINDER, graduated, 500 ml	PHEN	1	
CYLINDER, graduated, 100 ml	DO (W), DO (M), BOD GC & ECH COD SUR	1 1 1 1	
CYLINDER, graduated, 50 ml	KD & ACC COD PHEN OG	1 2 1 1	
CYLINDER, graduated, 10 ml	DO (W), DO (M), BOD OG SUR	1 1 1	
DESICCATOR(s)	GC & ECH OG OG	See Remarks See Remarks See Remarks	To hold 1 150 ml beaker per station. To hold 1 25 or 50 ml beaker per station. To hold 1 125 ml distilling flask per station.
DIGESTION RACK	KD	See Remarks	1 per 4 stations.
FILTER PAPER, medium porosity.	OG SUR	1 4	
FLASK, distilling, 24/40 ground glass neck.	OG	1	

DESCRIPTION	TEST	QUANTITY PER STATION	REMARKS
FLASK, Erlenmeyer, 1 liter.	GC & ECH PHEN	1 1	
FLASK, Erlenmeyer, 500 ml	DO (W), DO (M), BOD	1	
FLASK, Erlenmeyer, 250-500 ml, 24/40 ground glass neck.	COD	1	Part of reflux apparatus.
FLASK, Erlenmeyer, wide mouth, 250-500 ml.	COD	1	
FLASK, Erlenmeyer, 125 ml.	PHEN SUR	4 4	
FLASK, Kjeldahl, micro.	KD	1	
FLASK, round bottom, 24/40 ground glass long neck (SGA JF-2130 or equivalent).	PHEN	1	Part of distillation apparatus.
FLASK, volumetric, 100 ml	PHEN SUR	4 4	
FUNNEL	PHEN	1	To fit hole in offset adapter used in phenol distillation apparatus.
FUNNEL, powder, about 50 mm	GC & ECH	1	
FUNNEL, separatory, 1 liter.	GC & ECH OG	1 1	Teflon stopcock Teflon stopcock
FUNNELS, separatory, 200-250 ml.	SUR	4	Teflon stopcock
FUNNEL, short stem, about 50 mm.	DO (W), BOD COD OG SUR	1 1 1 1	To fit into a buret. To fit into a buret.

DESCRIPTION	TEST	QUANTITY PER STATION	REMARKS
GAS, carrier, 95% argon-methane	PCB GC & ECH	See Remarks See Remarks	1 cylinder per gas chromatograph. 1 cylinder per gas chromatograph.
GAS chromatograph	GC & ECH PCB	See Remarks See Remarks	1 per 2 stations. 1 per 2 stations.
GLASSES, safety	All	2	
GLASS beads (or equivalent)	PHEN KD COD	6-10 10 6-10	For use with distillation apparatus. For use with micro Kjeldahl flask. For use with reflux apparatus.
GLASS rod (about 6 inches)	OG	1	
GLASS wool	SUR	4 small pieces	
GLOVES, asbestos	KD & ACC	See Remarks	1 pair per steam distilliation apparatus.
HOT plate	COD	1	Large enough to accommodate a 500 ml Erlenmeyer flask. Part of reflux apparatus.
KUDERNA-DANISH apparatus (including a stopper for the cup)	GC & ECH	1	
MATCHES	DK & ACC PHEN	See Remarks See Remarks	Several per steam distillation apparatus. Several per distillation apparatus.
MEDICINE dropper	ECH	1	
MIXER, plunger-type (or long stirring rod)	BOD	1	For use with a 1 liter graduated cylinder.
OVEN, muffle, 400°C	GC & ECH		
PAPER, graph, linear	GC & ECH	See Remarks	1 sheet per gas chromatograph.

DESCRIPTION	TEST	QUANTITY PER STATION	REMARKS
PENCIL, grease (or other marking)	All	1	
pH METER & electrode, with pH 9, 10, or 11 buffer	PHEN	See Remarks	1 or 2 per class.
pH PAPER, sensitive at pH 2	OG	2	
PIPET bulb	DO (W), BOD KD & ACC KD COD PHEN SUR	1	
PIPET, volumetric, 20 ml	DO (W), BOD COD	1 1	
PIPET, volumetric, 5 ml	PHEN	1	
PIPET, volumetric, 2 ml	PHEN	2	
PIPET, volumetric 1 ml	BOD KD & ACC	See Remarks 1	Set of 4, per station.
PIPETS, volumetric or graduated	PHEN SUR	See Remarks See Remarks	To prepare the assigned standards. To prepare the assigned standards.
PIPET, volumetric or graduated, 2 ml	BOD	3	
PIPET, graduated, 10 ml	DO (W), BOD KD & ACC KD COD	1 2 1 1	
RACK (or large beaker)	KD & ACC	1. See Remarks	To hold the Nessler tubes.

DESCRIPTION	TEST	QUANTITY PER STATION	REMARKS
RING	GC & ECH	1	To support 1 liter separatory funnel.
	OG	1	To support 1 liter separatory funnel.
	PHEN	1	To support a 1 liter round bottom flask.
	SUR	4	To support 200-250 ml separatory funnels.
RING stand	DO (W), BOD	1	To support buret and clamp.
	GS & ECH	1	To support 1 liter separatory funnel and ring, and Kuderna-Danish apparatus.
	PHEN	1	
	OG	1	To support a ring and 1 liter separatory funnel.
	SUR	4	To support rings and 200-250 ml separatory funnels.
RULER, 12 inch	GC & ECH	See Remarks	1 per gas chromatograph.
	PCB	See Remarks	1 per gas chromatograph.
SIPHON	BOD	1	Long enough for use with a 1 liter graduated cylinder.
SPATULA, scoop-type	DO (W)	See Remarks	1 per trip balance.
SPECTROPHOTOMETER (Spectronic 20, or equivalent)	KD & ACC	See Remarks	1 per 4 stations.
	PHEN	See Remarks	1 per 4 stations.
	SUR	See Remarks	1 per 4 stations.
STEAM (or hot water) bath, 70° C and 80° C	OG	See Remarks	To accommodate 1 125 ml distilling flask per station.
STEAM (or hot water) bath	GC & ECH	See Remarks	To hold 1 Kuderna-Danish apparatus per station.
STEAM distillation apparatus, Micro-Kjeldahl	KD & ACC	1. See Remarks	1 per 6 stations, commercially available.
STOPPER, rubber (must be clean)	KD & ACC	See Remarks	To place over the end of the Nessler tube while shaking.

DESCRIPTION	TEST	QUANTITY PER STATION	REMARKS
STOPWATCH (or equivalent)	GC & ECH	See Remarks	1 per gas chromatograph.
	PCB	See Remarks	1 per gas chromatograph.
SUCTION device with pressure tubing	OG	1	See diagram on page 24-5
SYRINGE, micro, 50 or 100 μl	CA	See Remarks	1 per carbon analyzer.
SYRINGE, micro, 10 μl	GC & ECH	See Remarks	1 per gas chromatograph.
SYRINGE, micro, 5, 10 and 50 μl	GC & ECH	See Remarks	1 set per gas chromatograph.
TISSUES	SP	1 or 2, See Remarks	For wiping fingerprints from the spectrophotometer cell.
	CA	Several	
	GC & ECH	See Remarks	Several per gas chromatograph.
	PCB	See Remarks	Several per gas chromatograph.
	SUR	See Remarks	For wiping fingerprints from the spectrophotometer cell.
TONGS, crucible (or tissues)	OG	1. See Remarks	To handle the distilling flask.
TUBE, Nessler, 50 ml	KD & ACC	2	
	KD	1	
TOWELS, paper (or sponge)	All	Several	
WEIGHING boat, disposable, 1 or 2 inch square	DO (W), DO (M), BOD	1	
WIRE gauze	PHEN	1	Part of distillation apparatus.

Laboratory Reagent Requirements
(Compiled from section X of the IPW's)

DESCRIPTION	QUANTITY PER STATION	REMARKS
CARBON ANALYSES		
Potassium acid phthalate, 1 mg C per liter - 1000 ml 2.0424 g		Prepare 5 standards in the range of 0-100 mg C per liter, by diluting this solution. Approximately 10 ml of each.
Sample		Wastewater treatment plant effluent. Approximately 50 ml.
Water, distilled		To prepare solutions.
CHEMICAL OXYGEN DEMAND		
Ferroin indicator - 1000 ml 14.85 g 1, 10 - phenanthroline 6.95 g ferrous sulfate heptahydrate	0.5 ml	
Ferrous ammonium sulfate - 1000 ml 39 g ferrous ammonium sulfate hexahydrate 20 ml concentrated sulfuric acid	50 ml	
Ice		To fill 1 pan for every 2 stations.
Mercuric sulfate	0.4 g	
Potassium dichromate, 0.250 N - 1000 ml 12.259 g potassium dichromate	20 ml	
Sample	20 ml	wastewater treatment plant influent or effluent, as assigned.

DESCRIPTION	QUANTITY PER STATION	REMARKS
Sulfuric acid, concentrated	33 ml	
Sulfuric acid - silver sulfate 22 g silver sulfate per 9 lb. bottle sulfuric acid	27 ml	
Water, distilled		To prepare solutions.
DISSOLVED OXYGEN - WINKLER DISSOLVED OXYGEN - METER BIOCHEMICAL OXYGEN DEMAND		
Alkaline - iodide - azide 500 g sodium hydroxide or 700 g potassium hydroxide	4 ml	
Calcium chloride - 1000 ml 27.5 g anhydrous	1 ml	
Ferric chloride - 1000 ml 0.25 g	1 ml	
Manganous sulfate - 1000 ml 480 g manganous sulfate tetrahydrate, or 400 g manganous sulfate dihydrate, or 364 g manganous sulfate monohydrate	4 ml	
Magnesium sulfate - 1000 ml 22.5 g magnesium sulfate heptahydrate	1 ml	
Phosphate buffer - 1000 ml 8.5 g potassium dihydrogen phosphate 21.75 g dipotassium hydrogen phosphate 33.4 g disodium hydrogen phosphate heptahydrate 1.7 g ammonium chloride	1 ml	

DESCRIPTION	QUANTITY PER STATION	REMARKS
Potassium biiodate, 0.0375 N - 1000 ml 4.873 g	20 ml	
Potassium iodide	3 g	
Sample	As assigned	Wastewater treatment plant influent or effluent.
Starch - 1000 ml 10 g	4 ml	
Sodium thiosulfate, 0.75 N, stock - 1000 ml 186.15 g sodium thiosulfate pentahydrate		
Sodium thiosulfate, approximately 0.0375 N - 1000 ml 50 ml of sodium thiosulfate stock	45 ml	
Sulfuric acid, concentrated - 1000 ml	4 ml	
Sulfuric acid, 10% - 1000 ml 100 ml concentrated sulfuric acid	10 ml	
Water, distilled		To prepare solutions.
EXTRACTION OF CHLORINATED HYDROCARBONS		
n- hexane, pesticide grade		About 10 ml in a glass stoppered bottle at each gas chromatograph.
Methylene chloride, 15% in n- hexane (both pesticide grade)	200 ml	
Pesticide standards (see next page)		About 10 ml of A, of B, and of C in separate small glass stoppered bottles at each gas chromatograph.

DESCRIPTION	QUANTITY PER STATION	REMARKS
A. 0.05 ng lindane per µl 0.05 ng aldrin per µl 0.05 ng heptachlor epoxide per µl B. 0.10 ng lindane per µl 0.10 ng aldrin per µl 0.10 ng heptachlor expoxide per µl C. 0.15 ng lindane per µl 0.15 ng aldrin per µl 0.15 ng heptachlor epoxide per µl		
Sodium sulfate, anhydrous, granular	About 60 g	In 150 ml beaker in a desiccator. Previously muffled at 400°C for 4 hours.
Water, distilled	500 ml	In a 1 liter separatory funnel at each station, and spiked for use at the sample as indicated in VIII A. 5. of the IPW.
KJELDAHL DIGESTION		
Digestion reagent - 1000 ml 134 g potassium sulfate 200 ml concentrated sulfuric acid	10 ml	
Sample, synthetic, 0.065 mg ammonia nitrogen per 50 ml - 1000 ml	50 ml	See section X of the IPW, Laboratory-Kjeldahl Digestion, for preparation.
Water, distilled, ammonia-free		To prepare solutions.
KJELDAHL DISTILLATION AND AMMONIA CALIBRATION CURVE		
Ammonium chloride, stock, 1 mg ammonia nitrogen per ml - 1000 ml 3.819 g ammonium chloride		
Ammonium chloride, standard, 0.01 mg ammonia nitrogen per ml - 1000 ml 10 ml of ammonium stock	Maximum of 23 ml	Actual volume depends on standards assigned.

DESCRIPTION	QUANTITY PER STATION	REMARKS
Boric acid, 2% - 1000 ml 20 g boric acid	5 ml	Located at the distillation apparatus.
Nessler reagent - 1000 ml 100 g mercuric iodide 70 g potassium iodide 160 g sodium hydroxide	4 ml	
Sodium hydroxide - sodium thiosulfate - 1000 ml 500 g sodium hydroxide 25 g sodium thiosulfate pentahydrate	10 ml	Located at the distillation apparatus.
Water, distilled, ammonia-free		To prepare solutions.
OIL AND GREASE		
Extracting solvent	95 ml	Freon TF (DuPont), Genosolv D (Allied), or Genetron 113 (Allied).
Hydrochloric (or sulfuric) acid, 50%	5 ml	
Sample	1000 ml	Wastewater treatment plant effluent.
Sodium sulfate, anhydrous	1 g	
Water, distilled		To prepare solutions.
PHENOL		
Ammonium chloride - 1000 ml 50 g ammonium chloride	8 ml	
4 - aminoantipyrine - 1000 ml 20 g 4 - aminoantipyrine	8 ml	

DESCRIPTION	QUANTITY PER STATION	REMARKS
Buffer, pH 9, 10, or 11	100 ml	
Copper Sulfate - 1000 ml 100 g copper sulfate pentahydrate	5 ml	
Methyl Orange - 1000 ml 0.5 g methyl orange	3 drops	Eyedropper bottle.
Phenol, stock - 1000 ml 1 g phenol		
Phenol, intermediate standard, 10 µg phenol per ml - 1000 ml 10 ml of phenol stock	Maximum of 65 ml.	Actual volume depends on standards assigned.
Phosphoric acid - 1000 ml 100 ml of 85% phosphoric acid	1 ml	Eyedropper bottle.
Potassium ferricyanide - 1000 ml 80 g potassium ferricyanide	8 ml	
Sample	500 ml	Wastewater treatment plant effluent.
Water, distilled		To prepare solutions.
POLYCHLORINATED BIPHENYLS		
Aroclor 0.1 ng per µl		About 5 ml in a small glass stoppered bottle at each gas chromatograph.
p, p' - DDE 0.1 ng per µl		About 5 ml in a small glass stoppered bottle at each gas chromatograph.

DESCRIPTION	QUANTITY PER STATION	REMARKS
n - hexane, pesticide grade		About 10 ml in a small glass stoppered bottle at each gas chromatograph.
SURFACTANTS		
Chloroform	440 ml	
Linear alkylate sulfonate (LAS)		Sealed ampule available on written request from US EPA, EMSL, Cincinnati, Ohio 45268.
Linear alkylate sulfonate, standard, 10 μg 1 liter - 1000 ml 10 ml of ampule LAS per liter	Maximum of 40 ml	Actual volume depends on standards assigned.
Methylene blue (concentrate) - 1000 ml 1 g methylene blue		Solution is used in the diluted form.
Methylene blue (diluted) - 1000 ml 30 ml concentrate (see above) 6.8 ml concentrated sulfuric acid 50 g sodium dihydrogen phosphate monohydrate	100 ml	
Phenalphthalein - 1000 ml 5 g phenolphthalein	1 ml	Eyedropper bottle.
Sample	100 ml	Wastewater treatment plant effluent. A synthetic sample, prepared from the LAS standard may also be used.
Sodium Hydroxide, 1 N- 1000 ml 40 g sodium hydroxide	5 ml	Eyedropper bottle.
Sulfuric Acid, 1 N - 1000 ml 28 ml concentrated sulfuric acid	1 ml	Eyedropper bottle.

DESCRIPTION	QUANTITY PER STATION	REMARKS
Wash solution - 1000 ml 6.8 ml concentrated sulfuric acid 50 g sodium dihydrogen phosphate monohydrate	200 ml	
Water, distilled		To prepare solutions.

PART II - INSTRUCTIONAL PACKAGE WORKSHEETS

The Worksheets are for guidance to the Instructor who develops the subject matter covered in the course. These Worksheets are not scripts. The Instructor will need to make extensive and detailed preparation in order to perform the assigned tasks effectively and efficiently. The Instructional Packages do provide a perspective on the background of each analytical procedure, lesson-by-lesson learning achievement levels the students should attain, an indication of available audiovisual and other instructional resources, and an example course of action in pre-course preparation and classroom/laboratory instruction. The Instructor is free to modify the Worksheets to meet individual needs. It should be noted however, that associated additions and deletions will then be required in Sections V through XI of the Worksheet, and in Sections E and F of Part I.

Application of these Instructional Packages will help the Instructor to reduce the time required for planning and organizing a strategy of preparation and instruction. However, time and effort are required for physical preparations for classroom and laboratory instruction; time and effort are required for rehearsals of Instructor performance in classroom and laboratory. These requirements never can be met by such an Instructor's Guide as this; ultimately the Instructor is the key person in assuring that the student acquires the needed knowledge and skills.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Sample Handling - Field Through Laboratory

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 30 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: A basic element of quality assurance data is a reliable and valid sample, properly preserved, stored and documented. An analyst must be knowledgeable about sample handling to carry out his/her responsibilities.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of college inorganic chemistry, or equivalent. Although not essential, some experience in chemical laboratory work would aid comprehension/retention of information.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on the Subject Matter and have an overview of all the elements of a program to assure a valid, properly preserved sample whose integrity is documented from collection to storage.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, will have the use of the Training Manual, and a packet of information (VII A. 2. and 3.) regarding sample preservation.

C. Accepted Performance:

A grade of 70% on the quiz.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the Training Manual: Sample Handling - Field Through Laboratory.

2. Handout: The preservation procedures for samples collected for National Pollutant Discharge Elimination System or State Certification report purposes are to be finalized in the Federal Register sometime after January, 1981. To ensure that you distribute current information, contact your EPA Regional Quality Assurance Coordinator for copies of the appropriate information.

3. Handout: The preservation procedures specifically designed for drinking water samples have been published in the U.S. EPA Report No. EPA 600/8-78-008, May, 1978. A one page copy of the applicable information is page 7-4).

4. Optional - copies of standard reference books of methods listed in the outline (See VII A. 1. above) section, Methods of Analysis.

5. X-17, Sample Handling (8 slides); See XI, Description of Visual Materials (page 7-7).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Preview Available Media and prepare lesson, using the Sequencing below or your own organization of the material.

2. Reproduce the attached quiz; one copy for each participant, plus any desired file copies. See pages 14-4 and 14-5 ff.

3. Reproduce items VII A. 2. and 3.; one copy for each participant, plus any desired file copies.

B. Sequencing:

Classroom - discuss the sections in the Training Manual outline, Sample Handling - Field Through Laboratory, using X-17, Sample Handling.

1. Section I - Planning a Sample Program

   a. Factors to consider; X-17, 1. Define grab and composite samples; X-17, 2.

   b. Decisive criteria for making decisions should be incorporated into the discussion.

2. Section II - Representative Samples - Associated factors; X-17, 3.

3. Section III - Sample Identification - Topics for discussion; X-17, 4.

   Section III C - list of items

4. Section IV - Sample Container - Considerations for discussion; X-17, 5.

5. Section V - Sample Preservation - Functions; X-17, 6

   Section V C - Specific Methods - Note sources of information; General methods, X-17, 7.

   Section V D - Federal Register - Distribute handouts and discuss.

6. Section VI - Names of standard references for methods of analysis; preservation methods are usually included. Display a copy of each reference as it is mentioned.

Section VI E - Federal Register methodology. Note that there are regulations for NPDES, State certifications and Drinking Water. These are covered in detail in a separate lesson on Compliance Methodology.

7. Section VII - Order of Analyses - based on holding time factors; X-17, 8.

8. Destribute pages on preservation (See VII A. 3.). Stress theme of memo; i.e., that these are guidelines.

9. Record Keeping

   a. Field

   b. Laboratory

10. Section IX - Summary

Classroom - give the quiz and then read the correct answers from the attached answer sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

For Each Student:

Handout Materials

X. IPW REAGENT REQUIREMENTS:

None

From: EPA 600/8-78-008, May 1978 "Manual for Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies"

Table IV-2.—*Sample collecting, handling, and preservation*[1]

Parameter	Preservative[2]	Container[3]	Maximum holding time[4]
Arsenic	Conc HNO_3 to pH<2	P or G	6 months
Barium	Conc HNO_3 to pH<2	P or G	6 months
Cadmium	Conc HNO_3 to pH<2	P or G	6 months
Chromium	Conc HNO_3 to pH<2	P or G	6 months
Lead	Conc HNO_3 to pH<2	P or G	6 months
Mercury	Conc HNO_3 to pH<2	G P	38 days 14 days
Nitrate	Conc H_2SO_4 to pH<2	P or G	14 days
Selenium	Conc HNO_3 to pH<2	P or G	6 months
Silver	Conc HNO_3 to pH<2	P or G	6 months
Fluoride	None	P or G	1 month
Chlorinated hydrocarbons	Refrigerate at 4° C as soon as possible after collection	G with foil- or Teflon-lined cap	14 days[5]
Chlorophenoxys	Refrigerate at 4° C as soon as possible after collection	G with foil- or Teflon-lined cap	7 days[5]

---

[1] If a laboratory has no control over these factors, the laboratory director must reject any samples not meeting these criteria and so notify the authority requesting the analyses.

[2] If HNO_3 cannot be used because of shipping restrictions, sample may be initially preserved by icing and immediately shipping it to the laboratory. Upon receipt in the laboratory, the sample must be acidified with conc HNO_3 to pH<2. At time of analysis, sample container should be thoroughly rinsed with 1:1 HNO_3; washings should be added to sample.

[3] P = Plastic, hard or soft; G = Glass, hard or soft.

[4] In all cases, samples should be analyzed as soon after collection as possible.

[5] Well-stoppered and refrigerated extracts can be held up to 30 days.

NAME__________________________

QUIZ

Sample Handling - Field Through Laboratory

Check the correct answer or fill in the missing word(s) as indicated.

1. A "representative" sample represents

____ the best water quality area of the soruce at the time of sampling.

____ the conditions existing in the water quality of the source at the time of sampling.

____ the anticipated water quality area of the source at the time sampling.

____ the worst water quality area of the source at the time of sampling.

2. Written procedures to provide an accurate record for the chain-of-custody of monitoring samples should be developed for at least

____ sample collection and transport.

____ sample collection, transport and delivery to a laboratory.

____ sample collection, transport, delivery to a laboratory and distribution to analyst(s).

____ sample collection, transport, delivery to a laboratory, distribution to analyst(s) ans storage or disposal.

3. The first consideration when drawing up a time schedule for analyzing samples is

____ the preservative used.

____ the number of analysts available.

____ the holding time guideline for the constituent of interest.

____ the quality control checks to be used.

____ the number of samples to be processed.

4. Two materials from which sample containers my be fabricated are ________________ and ___________________.

5. Four items of useful identification information which may be included with a sample are _______________, _______________, _______________, and _______________.

NAME ________________________

QUIZ - ANSWER SHEET

Sample Handing - Field Through Laboratory

Check the correct answer or fill in the missing word(s) as indicated.

1. A "representative" sample represents

____ the best water quality area of the source at the time of sampling.

_X_ the conditions existing in the water quality of the source at the time of sampling.

____ the anticipated water quality area of the source at the time sampling.

____ the worst water quality area of the source at the time of sampling.

2. Written procedures to provide an accurate recrod for the chain-of-custody of monitoring samples should be developed for at least

____ sample collection and transport.

____ sample collection, transport and delivery to a laboratory.

____ sample collection, transport, delivery to a laboratory and distribution to analyst(s).

_X_ sample collection, transport, delivery to a laboratory, distribution to analyst(s) and storage or disposal.

3. The first consideration when drawing up a time schedule for analyzing samples is

____ the preservative used.

____ the number of analysts available.

_X_ the holding time guideline for the consitutent of interest.

____ the quality control checks to be used.

____ the number of samples to be processed.

4. Two materials from which sample containers may be fabricated are glass and plastic; or hard rubber.

5. Four items of useful identification information which may be included with a sample are sample code number, signature of sampler, signature of witness, and sampling location; sampling equipment, date of collection time of collection, type of sample (grab or composite), water temperature, sampling conditions (weather, water), preservative used (if any), record of determinations done in the field type of analyses to be done in the laboratory.

Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For "fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

XI. DESCRIPTION OF VISUAL MATERIALS

SERIES X-17: SAMPLE HANDLING

COLLECTION OF SAMPLES

- SAMPLING SITE
- EQUIPMENT
- QUANTITY
- TYPE
- PRESERVATION

SLIDE 1

TYPES OF SAMPLES

GRAB SAMPLE - INDICATES CONDITION AT TIME OF SAMPLING

COMPOSITE SAMPLE - INDICATES AVERAGE CONDITION OVER COMPOSITING PERIOD

SLIDE 2

REPRESENTATIVE SAMPLES

- CLEAN CONTAINER
- COMPOSITING SAMPLES
- PROPER EQUIPMENT
- SLUDGE OR MUD

SLIDE 3

SAMPLE IDENTIFICATION

- PROPER TAG
- USEFUL IDENTIFICATION INFORMATION
- SAMPLE CUSTODY

SLIDE 4

SAMPLE CONTAINERS

- AVAILABLE MATERIALS
- CHEMICAL AND PHYSICAL INTERACTIONS
- MAILING REQUIREMENTS PRELIMINARY CHECK
- CLEANING
- STORAGE

SLIDE 5

SAMPLE PRESERVATION

- RETARD BIOLOGICAL ACTION
- RETARD PRECIPITATION OR HYDROLYSIS
- REDUCE VOLATILITY

SLIDE 6

GENERAL METHODS OF SAMPLE PRESERVATION

- pH CONTROL
- CHEMICAL ADDITION
- REFRIGERATION OR
- FREEZING

SLIDE 7

TIME INTERVAL BETWEEN COLLECTION AND ANALYSIS

- DEPENDS ON -SAMPLE TYPE
  - STABILITY
  - STORAGE CONDITIONS

SLIDE 8 (LAST SLIDE)

SLIDE ______

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Compliance Methodology

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 60 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: A basic element of quality assurance for chemical analytical data is use of a recognized analytical procedure. In addition, federal regulations affect the final choice of methodology for compliance samples. An analyst must know about these regulations to fulfill the attendant responsibilities.

V. ENTRY LEVEL BEHAVIOR: No specific skills. Familiarity with fundamentals of chemical analytical methods would facilitate comprehension/retention of the information. Though not essential, it is orderly to complete the lesson on sample handling prior to this lesson.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will be able to use Federal Registers and pertinent US EPA explanatory memos to choose approved analytical methodology for compliance with the National Pollutant Discharge Elimination System (NPDES) and the National Interim Primary Drinking Water Regulations. Also, the participant will take a quiz on the Subject Matter.

B. Conditions:

He/she will have heard the lecture on the Subject Matter explaining the applicability and format of the items in Section VII A.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. The instructor will judge the participant's attention and use of the materials, as directed, during the discussion.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the Training Manual:

a. "Methodology for Chemical Analysis of Water and Wastewater" is a glossary of the methods promulgated for obtaining compliance data.

2. Handouts:

a. National Pollutant Discharge Elimination System - NPDES:

1) Guidelines Establishing Test Procedures, 40 CFR 136, Federal Register, 12/1/76, pp 52780-52786. A copy is at the end of this IPW and also an errata sheet. Changes were proposed 12/3/79, pp 69464-69575. These proposed regulations included publication for comment of GC, HPLC and GC/MS methods. The regulations should be finalized after January, 1981. Check with your EPA Regional Quality Assurance Coordinator about the status of these methods and regulations. (Name and phone number available from EMSL-Cinti, 513/684-7301). The Coordinator can supply appropriate copies to you.

2) Summary of protocols to apply for approval of an alternative test procedure for regulatory purposes. A copy is at the end of this IPW.

3) Federal Register notices of alternative methods approved to date (8/80) are at the end of this IPW. Contact your EPA Regional Quality Assurance Coordinator for copies of notices published since then.

4) EPA-EMSL memo on "Use of Chemical Test Kits for Compliance Monitoring". A copy is at the end of this IPW.

5) EPA-EMSL memo on "Use of Prepared Reagents in NPDES Compliance". A copy is at the end of this IPW.

b. National Interim Primary Drinking Water Regulations - NIPDWR:

1) NIPDWR, 40 CFR 141, Federal Register, 12/24/75, pp 59566-59574. A copy is at the end of this IPW.

2) Amendments to NIPDWR were proposed 7/19/79, 40 CFR 141. The amendments were finalized 40 CFR Part 141, Federal Register, 8/27/80, pp 57332-57346. Copies of pp 57344-57346 are at the end of this IPW. The pages contain the current (8/80), approved analytical requirements for drinking water analysis. These include an update of the approved methods presented in the 12/24/75 NIPDWR and also alternate analytical techniques approved by EPA since that date. Check with your EPA Regional Quality Assurance Coordinator to get copies of any additional, pertinent Federal Register notices published after 8/80.

3) The final rule for Trihalomethanes in drinking water (40 CFR 141) was published in the Federal Register, 11/29/79 on pp 68624-68642. Copies of page 68624 and pp 68641-68642 contain the final wording and are at the end of this IPW.

c. National Secondary Drinking Water Regulations - NSDWR:

1) NSDWR, 40 CFR 143, Federal Register, pp. 42198-42199. A copy is at the end of this IPW.

B. Suggested Media:

1. OPTIONAL: Copies of the methods manuals cited for NPDES and NIPDWR could be available for display/inspection.

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Contact your EPA Regional Quality Assurance Coordinator to check if any Federal Registers pertaining to NPDES or to NIPDWR have been issued since the ones referenced in VII A above (8/80). If so, obtain copies from the Coordinator for your class.

2. Thoroughly review all pertinent federal register information and handout materials. Since the regulations are legally binding, it is imperative to impart correct and current information to the participants.

3. Prepare the lesson using the Sequencing below or your own organization of the materials.

4. Duplicate copies of all the handout materials. Most can be copied back-to-back, but be careful to keep each handout as an entity so participants can use each independent of the other. Collate the handouts into a packet for each participant to minimize distribution time.

B. Sequencing:

1. Hand out the packets of information.

2. Introduction

   a. Choice of methodology affects validity and reliability of data.

   b. Analytical programs affected by federal legislation include those involving point source discharges (NPDES) and drinking water (NIPDWR).

3. National Pollutant Discharge Elimination System (NPDES). CAUTION: Use the current Federal Register. The NPDES Guidelines are scheduled for update after January, 1981.

   a. Applicability of the regulations. See 12/1/76, page 52780, paragraph 2.

   b. Overview of Table 1

1) Column 1: Alphabetical listing of parameters and units. Note subcategories.

2) Column 2: Method listings and possibly specification of a pretreatment and/or method choice, e.g., acidity end point is to be pH of 8.2. A summary of the principles involved in the listed methodology can be found in the training manual outline, "Methodology for Chemical Analysis of Water and Wastewater". You can use the outline to list the types of methodology found in the Table. Note that many automated methods are approved.

3) Remaining columns list sources (with page numbers) of approved methods. You can display copies of the cited manuals at this time and leave them to be available for inspection by participants.

4) Note the location (by number) of the parameters to be taught during the course. Also note the method to be used for course laboratory sessions.

5) Note any errata (handout).

c. Go over highlights of Applying for Approval of Alternative Test Procedures (handout).

1) Limited use

2) Nationwide use

d. Go through the Federal Register notices of alternative methods approved for nationwide use, briefly noting the contents.

e. Go over highlights of the memo on "Use of Chemical Test Kits for Compliance Monitoring" (handout).

f. Note highlights of the memo on "Use of "Prepared" Reagents in NDPES Compliance" (handout).

4. Drinking Water Regulations. CAUTION: Use the current Federal Registers. The 12/24/75 Federal Register is the current (8/80) National Interim Primary Drinking Water Regulation (NIPDWR), along with the Amendments to NIPDWR which were finalized 8/27/80.

a. Applicability of the regulations. See 12/24/75, page 59566, "Water Systems Covered" and page 595970, "Subpart A - General".

b. Maximum Contaminant Levels are listed in 12/24/75 for Chemicals on page 59570 and for Turbidity on page 59571.

c. The analytical methodology in the 12/24/75 regulation was updated and expanded in the 8/27/80 regulation. Therefore, use pp 57344-57346 from the 8/27/80 Federal Register to cite the location of the listings of approved methods: 141.22 Turbidity, 141.23 Inorganics, 141.24 Organics, 141.41 Sodium, 141.42 Corrosivity Characteristics. Also highlight section 141.28 Approved Laboratories, and section 141.27 Alternate Analytical Techniques. (Alternate methods approved between the original NIPDWR (12/24/75) and 8/80 are included in the sections just cited).

d. For Trihalomethanes, use the 11/29/79 Federal Register excerpts. The "Summary" on page 68624 gives an overview of the regulation. The maximum contaminant level is on page 68641, paragraph 141.12. The methodology is cited on page 68642, middle column, (1) and (2).

e. There are "National Secondary Drinking Water Regulations" in another part of the 7/19/79 Federal Register on pp 42198-42199. These represent reasonable goals for the aesthetic quality of drinking water but are not to be federally enforceable. Suggested Maximum Contaminants Levels and Analytical Methodology are presented on these pages.

5. A 10 minute discussion period should be scheduled later to answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. For each student:

1. Copies of handout materials

X. IPW REAGENT REQUIREMENTS:

A. None

NAME ________________________

QUIZ

Compliance Methodology

Check the correct answer or fill in the missing word(s) as indicated.

1. Three sources of analytical methods approved for NPDES are ______________, ________________, and ________________.

2. Kits may be substituted for an NPDES analytical method at the discretion of the analyst.

   ____ T

   ____ F

3. Purchased reagents may be utilized for an NPDES method if they have been prepared according to the approved method.

   ____ T

   ____ F

4. Automated methods for colorimetric determinations have not yet been approved for NPDES testing.

   ____ T

   ____ F

5. There is no provision to obtain approval of alternative analytical methods for drinking water analysis.

   ____ T

   ____ F

6. National Secondary Drinking Water Regulations control contaminants that affect the aesthetic quality of drinking water.

   ____ T

   ____ F

NAME ______________________

QUIZ - ANSWER SHEET

Compliance Methodology

Check the correct answer or fill in the missing word(s) as indicated.

1. Three sources of analytical methods approved for NPDES are Standard Methods, ASTM, and Methods for Chemical Analysis of Water & Wastes, or EPA Methods Manual or USGS.

2. Kits may be substituted for an NPDES analytical method at the discretion of the analyst.

   ___ T
   X F

3. Purchased reagents may be utlized for an NPDES method if they have been prepared according to the approved method.

   X T
   ___ F

4. Automated methods for colorimetric determinations have not yet been approved for NPDES testing.

   ___ T
   X F

5. There is no provision to obtain approval of alternative analytical methods for drinking water analysis.

   ___ T
   X F

6. National Secondary Drinking Water Regulations control contaminants that affect the aesthetic quality of drinking water.

   X T
   ___ F

Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For "fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

federal register

WEDNESDAY, DECEMBER 1, 1976

THE NATIONAL ARCHIVES OF THE UNITED STATES 1934

PART II:

ENVIRONMENTAL PROTECTION AGENCY

■

WATER PROGRAMS

Guidelines Establishing Test Procedures for the Analysis of Pollutants

Amendments

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Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY

SUBCHAPTER D—WATER PROGRAMS

[FRL 630-4]

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Amendment of Regulations

On June 9, 1975, proposed amendments to the Guidelines Establishing Test Procedures for the Analysis of Pollutants (40 CFR 136) were published in the FEDERAL REGISTER (40 FR 24535) as required by section 304(g) of the Federal Water Pollution Control Act Amendments of 1972 (86 Stat. 816, et seq., Pub. L. 92–500, 1972) hereinafter referred to as the Act.

Section 304(g) of the Act requires that the Administrator shall promulgate guidelines establishing test procedures for the analysis of pollutants that shall include factors which must be provided in: (1) any certification pursuant to section 401 of the Act, or (2) any permit application pursuant to section 402 of the Act. Such test procedures are to be used by permit applicants to demonstrate that effluent discharges meet applicable pollutant discharge limitations and by the States and other enforcement activities in routine or random monitoring of effluents to verify compliance with pollution control measures.

Interested persons were requested to submit written comments, suggestions, or objections to the proposed amendments by September 7, 1975. One hundred and thirty-five letters were received from commenters. The following categories of organizations were represented by the commenters: Federal agencies accounted for twenty-four responses; State agencies accounted for twenty-six responses; local agencies accounted for seventeen responses; regulated major dischargers accounted for forty-seven responses; trade and professional organizations accounted for eight responses; analytical instrument manufacturers and vendors accounted for seven responses; and analytical service laboratories accounted for six responses.

All comments were carefully evaluated by a technical review committee. Based upon the review of comments, the following principal changes to the proposed amendments were made:

(A) *Definitions.* Section 136.2 has been amended to update references: Twenty commenters, representing the entire spectrum of responding groups pointed out that the references cited in §§ 136.2(f), 136.2(g), and 136.2(h) were out-of-date; §§ 136.2(f), 136.2(g), and 136.2(h), respectively, have been amended to show the following editions of the standard references: "14th Edition of Standard Methods for the Examination of Water and Waste Water;" "1974 EPA Manual of Methods for the Analysis of Water and Waste;" and "Part 31, 1975 Annual Book of ASTM Standards."

(B) *Identification of Test Procedures.* Both the content and format of § 136.3, "Table I, List of Approved Test Procedures" have been revised in response to twenty-one comments received from State and local governments, major regulated dischargers, professional and trade associations, and analytical laboratories.

Table I has been revised by:

(1) The addition of a fourth column of references which includes procedures of the United States Geological Survey which are equivalent to previously approved methods.

(2) The addition of a fifth column of miscellaneous references to procedures which are equivalent to previously approved methods.

(3) Listing generically related parameters alphabetically within four subcategories: bacteria, metals, radiological and residue, and by listing these subcategory headings in alphabetic sequence relative to the remaining parameters.

(4) Deleting the parameter "Algicides" and by entering the single relevant algicide, "Pentachlorophenol" by its chemical name.

(C) *Clarification of Test Parameters.* The conditions for analysis of several parameters have been more specifically defined as a result of comments received by the Agency:

(1) In response to five commenters representing State or local governments, major dischargers, or analytical instrument manufacturers, the end-point for the alkalinity determination is specifically designated as pH 4.5.

(2) Manual digestion and distillation are still required as necessary preliminary steps for the Kjeldahl nitrogen procedure. Analysis after such distillation may be by Nessler color comparison, titration, electrode, or automated phenolate procedures.

(3) In response to eight commenters representative of Federal and State governments, major dischargers, and analytical instrument manufacturers, manual distillation at pH 9.5 is now specified for ammonia measurement.

(D) *New Parameters and Analytical Procedures.* Forty-four new parameters have been added to Table I. In addition to the designation of analytical procedures for these new parameters, the following modifications have been made in analytical procedures designated in response to comments.

(1) The ortho-tolidine procedure was not approved for the measurement of residual chlorine because of its poor accuracy and precision. Its approval had been requested by seven commenters representing major dischargers, State, or local governments, and analytical instrument manufacturers. Instead, the N,N-diethyl-p-phenylenediamine (DPD) method is approved as an interim procedure pending more intensive laboratory testing. It has many of the advantages of the ortho-tolidine procedure such as low cost, ease of operation, and also is of acceptable precision and accuracy.

(2) The Environmental Protection Agency concurred with the American Dye Manufacturers' request to approve its procedure for measurement of color, and copies of the procedure are now available at the Environmental Monitoring and Support Laboratory, Cincinnati (EMSL–CI)

(3) In response to three requests from Federal, State governments, and dischargers, "hardness," may be measured as the sum of calcium and magnesium analyzed by atomic absorption and expressed as their carbonates.

(4) The proposal to limit measurement of fecal coliform bacteria in the presence of chlorine to only the "Most Probable Number" (MPN) procedure has been withdrawn in response to requests from forty-five commenters including State pollution control agencies, permit holders, analysts, treatment plant operators, and a manufacturer of analytical supplies. The membrane filter (MF) procedure will continue to be an approved technique for the routine measurement of fecal colifiorm in the presence of chlorine. However, the MPN procedure must be used to resolve controversial situations. The technique selected by the analyst must be reported with the data.

(5) A total of fifteen objections, representing the entire spectrum of commenters, addressed the drying temperatures used for measurement of residues. The use of different temperatures in drying of total residue, dissolved residue and suspended residue was cited as not allowing direct intercomparability between these measurements. Because the intent of designating the three separate residue parameters is to measure separate waste characteristics (low drying temperatures to measure volatile substances, high drying temperatures to measure anhydrous inorganic substances), the difference in drying temperatures for these residue parameters must be preserved.

(E) *Deletion of Measurement Techniques.* Some measurement techniques that had been proposed have been deleted in response to objections raised during the public comment period.

(1) The proposed infrared spectrophotometric analysis for oil and grease has been withdrawn. Eleven commenters representing Federal or State agencies and major dischargers claimed that this parameter is defined by the measurement procedure. Any alteration in the procedure would change the definition of the parameter. The Environmental Protection Agency agreed.

(2) The proposed separate parameter for sulfide at concentrations below 1 mg/l, has been withdrawn. Methylene blue spectrophotometry is now included in Table I as an approved procedure for sulfide analysis. The titrimetric iodine procedure for sulfide analysis may only be used for analysis of sulfide at concentrations in excess of one milligram per liter.

(F) *Sample Preservation and Holding Times.* Criteria for sample preservation and sample holding times were requested by several commenters. The reference for sample preservation and holding time criteria applicable to the Table I parameters is given in footnote (1) of Table I.

(G) *Alternate Test Procedures.* Comments pertaining to § 136.4, Application for Alternate Test Procedures, included objections to various obstacles within these procedures for expeditious approval of alternate test procedures. Four analytical instrument manufacturers commented that by limiting of application for review and/or approval of alternate test procedures to NPDES permit holders, § 136.4 became an impediment to the commercial development of new or improved measurement devices based on new measurement principles. Applications for such review and/or approval will now be accepted from any person. The intent of the alternate test procedure is to allow the use of measurement systems which are known to be equivalent to the approved test procedures in waste water discharges.

Applications for approval of alternate test procedures applicable to specific discharges will continue to be made only by NPDES permit holders, and approval of such applications will be made on a case-by-case basis by the Regional Administrator in whose Region the discharge is made.

Applications for approval of alternate test procedures which are intended for nationwide use can now be submitted by any person directly to the Director of the Environmental Monitoring and Support Laboratory in Cincinnati. Such applications should include a complete methods write-up, any literature references, comparability data between the proposed alternate test procedure and those already approved by the Administrator. The application should include precision and accuracy data of the proposed alternate test procedure and data confirming the general applicability of the test procedure to the industrial categories of waste water for which it is intended. The Director of the Environmental Monitoring and Support Laboratory, after review of submitted information, will recommend approval or rejection of the application to the Administrator, or he will return the application to the applicant for more information. Approval or rejection of applications for test procedures intended for nationwide use will be made by the Administrator, after considering the recommendation made by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati. Since the Agency considers these procedures for approval of alternate test procedures for nationwide use to be interim procedures, we will welcome suggestions for criteria for approval of alternate test procedures for nationwide use. Interested persons should submit their written comments in triplicate on or before June 1, 1977 to: Dr. Robert B. Medz, Environmental Protection Technologist, Monitoring Quality Assurance Standardization, Office of Monitoring and Technical Support (RD-680), Environmental Protection Agency, Washington, D.C. 20460.

(H) *Freedom of Information.* A copy of all public comments, an analysis by parameter of those comments, and documents providing further information on the rationale for the changes made in the final regulation are available for inspection and copying at the Environmental Protection Agency Public Information Reference Unit, Room 2922, Waterside Mall, 401 M Street, SW., Washington, D.C. 20460, during normal business hours. The EPA information regulation 40 CFR 2 provides that a reasonable fee may be charged for copying such documents.

Effective date: These amendments become effective on April 1, 1977.

Dated: November 19, 1976.

JOHN QUARLES,
Acting Administrator,
Environmental Protection Agency.

Chapter I, Subchapter D, of Title 40, Code of Federal Regulations is amended as follows:

1. In § 136.2, paragraphs (f), (g), and (h) are amended to read as follows:

§ 136.2 Definitions.

* * * * *

(f) "Standard Methods" means *Standard Methods for the Examination of Water and Waste Water,* 14th Edition, 1976. This publication is available from the American Public Health Association, 1015 18th Street, N.W., Washington, D.C. 20036.

(g) "ASTM" means *Annual Book of Standards, Part 31, Water, 1975.* This publication is available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

(h) "EPA Methods" means *Methods for Chemical Analysis of Water and Waste, 1974.* Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, Cincinnati, Ohio 45268; U.S. Environmental Protection Agency, Office of Technology Transfer, Industrial Environmental Research Laboratory, Cincinnati, Ohio 45268. This publication is available from the Office of Technology Transfer.

2. In § 136.3, the second sentence of paragraph (b) is amended, and a new paragraph (c) is added to read as follows:

§ 136.3 Identification of test procedures.

* * * * *

(b) * * * Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator or the Director upon the recommendation of the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(c) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring and Support Laboratory, Cincinnati, additional alternate test procedures for nationwide use.

3. Table I of § 136.3 is revised by listing the parameters alphabetically; by adding 44 new parameters; by adding a fourth column under references listing equivalent United States Geological Survey methods; by adding a fifth column under references listing miscellaneous equivalent methods; by deleting footnotes 1 through 7 and adding 24 new footnotes, to read as follows:

* * * * *

TABLE I.—*List of approved test procedures*[1]

Parameter and units	Method	References (page nos.) 1974 EPA methods	14th ed. standard methods	Pt. 31 1975 ASTM	USGS methods[2]	Other approved methods
1. Acidity, as $CaCO^3$, milligrams per liter.	Electrometric end point (pH of 8.2) or phenolphthalein end point.	1	273(4d)	116	40	[8](607)
2. Alkalinity, as $CaCO^3$, milligrams per liter.	Electrometric titration (only to pH 4.5) manual or automated, or equivalent automated methods.	3 5	278	111	41	[8](607)
3. Ammonia (as N), milligrams per liter.	Manual distillation[4] (at pH 9.5) followed by nesslerization, titration, electrode, Automated phenolate.	159 165 168	410 412 616	337	116	[8](614)
BACTERIA						
4. Coliform (fecal)[5], number per 100 ml.	MPN;[6] membrane filter.		922 937		[7](45)	
5. Coliform (fecal)[5] in presence of chlorine, number per 100 ml.	do.[6][7]		922 928, 937			
6. Coliform (total),[5] number per 100 ml.	do.[6]		916 928		[7](35)	
7. Coliform (total)[5] in presence of chlorine, number per 100 ml.	MPN;[6] membrane filter with enrichment.		916 933			
8. Fecal streptococci,[5] number per 100 ml.	MPN;[6] membrane filter; plate count.		943 944 947		[7](50)	
9. Benzidine, milligrams per liter.	Oxidation—colorimetric[9]					
10. Biochemical oxygen demand, 5-d (BOD_5), milligrams per liter.	Winkler (Azide modification) or electrode method.		543		[7](50)	[10](17)
11. Bromide, milligrams per liter.	Titrimetric, iodine-iodate.	14		323	58	
12. Chemical oxygen demand (COD), milligrams per liter.	Dichromate reflux.	20	550	472	124	[8](610) [10](17)
13. Chloride, milligrams per liter.	Silver nitrate; mercuric nitrate; or automated colorimetric-ferricyanide.	29 31	303 304 613	267 265	[11](46)	[8](615)

See footnotes at end of table.

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods [9]	
14. Chlorinated organic compounds (except pesticides), milligrams per liter.	Gas chromatography [11]					
15. Chlorine—total residual, milligrams per liter.	Iodometric titration, amperometric or starch-iodine end-point; DPD colorimetric or Titrimetric methods (these last 2 are interim methods pending laboratory testing).	35	318 322 332 329	278		
16. Color, platinum cobalt units or dominant wave length, hue, luminance, purity.	Colorimetric; spectrophotometric; or ADMI procedure.[13]	36 39	64 66		82	
17. Cyanide, total,[14] milligrams per liter.	Distillation followed by silver nitrate titration or pyridine pyrazolone (or barbituric acid) colorimetric.	40	361	503	85	[10](22)
18. Cyanide amenable to chlorination, milligrams per liter.	do.	40	376	505		
19. Dissolved oxygen, milligrams per liter.	Winkler (Azide modification) or electrode method.	51 56	443 450	368	126	[8](600)
20. Fluoride, milligrams per liter.	Distillation [4] followed by ion electrode; SPADNS; or automated complexone.	65 59 61	389 391 393 614	307 305	93	
21. Hardness—Total, as $CaCO_3$, milligrams per liter.	EDTA titration; automated colorimetric; or atomic absorption (sum of Ca and Mg as their respective carbonates).	68 70	202	161	94	[8](617)
22. Hydrogen ion (pH), pH units.	Electrometric measurement.	239	460	178	129	[8](606)
23. Kjeldahl nitrogen (as N), milligrams per liter.	Digestion and distillation followed by nesslerization, titration, or electrode; automated digestion automated phenolate.	175 165 182	437		122	[8](612)
METALS						
24. Aluminum—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Eriochrome Cyanine R).	92	152 171		[11] (19)	
25. Aluminum—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced methods for total aluminum.					
26. Antimony—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	94				
27. Antimony—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total antimony.					
28. Arsenic—Total, milligrams per liter.	Digestion followed by silver diethyldithiocarbamate; or atomic absorption.[16] [18]	9 95	285 283 159		[11] (31) [11] (37)	
29. Arsenic—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total arsenic.					
30. Barium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	97	152		52	
31. Barium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total barium.					
32. Beryllium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Aluminon).	99	152 177		53	
33. Beryllium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total beryllium.					
34. Boron—Total, milligrams per liter.	Colorimetric (Curcumin).	13	287			
35. Boron—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total boron.					
36. Cadmium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Dithizone).	101	148 182	345	62	[8] (619) [10] (37)
37. Cadmium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total cadmium.					
38. Calcium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption; or EDTA titration.	103	148 189	345	66	
39. Calcium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total calcium.					
40. Chromium VI, milligrams per liter.	Extraction and atomic absorption; colorimetric (Diphenylcarbazide).	89, 105	192		[8]76 75	
41. Chromium VI—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for chromium VI.					
42. Chromium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Diphenylcarbazide).	105	148 192	345 286	78 77	[8] (619)
43. Chromium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total chromium.					

See footnotes at end of table.

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.): Pt. 31 1975 ASTM	References (page nos.): USGS methods [9]	Other approved methods
44. Cobalt—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18]	107	148	345	80	[10] (37)
45. Cobalt—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total cobalt.					
46. Copper—Total, milligrams per liter.	Digestion [16] followed by atomic absorption [18] or by colorimetric (Neocuproine).	108	148 196	345 203	83	[3] (619) [10] (37)
47. Copper—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total copper.					
48. Gold—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18]					
49. Iridium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18]					
50. Iron—Total, milligrams per liter.	Digestion [16] followed by atomic absorption [18] or by colorimetric (Phenanthroline).	110	148 208	345 326	102	[3] (619)
51. Iron—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total iron.					
52. Lead—Total, milligrams per liter.	Digestion [16] followed by atomic absorption [18] or by colorimetric (Dithizone).	112	148 215	345	105	[3] (619)
53. Lead—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total lead.					
54. Magnesium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption; or gravimetric.	114	148 221	345	109	[3] (619)
55. Magnesium—Dissolved milligrams per liter.	0.45 micron filtration [17] dollowed by referenced method for total magnesium.					
56. Manganese—Total milligrams per liter.	Digestion [16] followed by atomic absorption [18] or by colorimetric (Persulfate or periodate).	116	148 225, 227	345	111	[3] (619)
57. Manganese—Dissolved milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total manganese.					
58. Mercury—Total, milligrams per liter.	Flameless atomic absorption.	118	156	338	[14] (51)	
59. Mercury—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total mercury.					
60. Molybdenum—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18]	130		350		
61. Molybdenum—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total molybdenum.					
62. Nickel—Total, milligrams per liter.	Digestion [16] followed by atomic absorption [18] or by colorimetric (Heptoxime).	141	148	345	115	
63. Nickel—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total nickel.					
64. Osmium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18]					
65. Palladium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18]					
66. Platinum—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18]					
67. Potassium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption, colorimetric (Cobaltinitrite), or by flame photometric.	143	 285 284	 403	134	[3] ([illegible])
68. Potassium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced meth-of for total potassium.					
69. Rhodium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18]					
70. Ruthenium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18]					
71. Selenium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption.[18] [19]	145	159			
72. Selenium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total selenium.					
73. Silica—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by colorimetric (Molybdosilicate).	274	487	398	139	
74. Silver—Total,[20] milligrams per liter.	Digestion [16] followed by atomic absorption [18] or by colorimetric (Dithizone).	146	148 243		142	[3] (619) [10] (37)
75. Silver—Dissolved,[20] milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total sliver.					
76. Sodium—Total, milligrams per liter.	Digestion [16] followed by atomic absorption or by flame photometric.	147	 250	 406	143	[3] (621)
77. Sodium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total sodium.					

See footnotes at end of table

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.) Pt. 31 1975 ASTM	References (page nos.) USGS methods [2]	Other approved methods
78. Thallium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	149				
79. Thallium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total thallium.					
80. Tin—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	150			[11] (65)	
81. Tin—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total tin.					
82. Titanium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption.[16]	151				
83. Titanium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total titanium.					
84. Vanadium—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Gallic acid).	152	152 260	441	[11] (67)	
85. Vanadium—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total vanadium.					
86. Zinc—Total, milligrams per liter.	Digestion [15] followed by atomic absorption [16] or by colorimetric (Dithizone).	155	148 265	345	159	[8] (619) [10] (37)
87. Zinc—Dissolved, milligrams per liter.	0.45 micron filtration [17] followed by referenced method for total zinc.					
88. Nitrate (as N), milligrams per liter.	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction.[21]	201 197 207	423 427 620	358	119	[8] (614) [10] (28)
89. Nitrite (as N), milligrams per liter.	Manual or automated colorimetric (Diazotization).	215	434		121	
90. Oil and grease, milligrams per liter.	Liquid-liquid extraction with trichloro-trifluoroethane-gravimetric.	229	515			
91. Organic carbon; total (TOC), milligrams per liter.	Combustion—Infrared method.[22]	236	532	467	[23] (4)	
92. Organic nitrogen (as N), milligrams per liter.	Kjeldahl nitrogen minus ammonia nitrogen.	175, 159	437		122	[8] (612, 614)
93. Orthophosphate (as P), milligrams per liter.	Manual or automated ascorbic acid reduction.	249 256	481 624	384	131	[8] (621)
94. Pentachlorophenol, milligrams per liter.	Gas chromatography [12]					
95. Pesticides, milligrams per liter.	do.[12]		555	529	[25] (24)	
96. Phenols, milligrams per liter.	Colorimetric, (4AAP)	241	582	545		
97. Phosphorus (elemental), milligrams per liter.	Gas chromatography [26]					
98. Phosphorus; total (as P), milligrams per liter.	Persulfate digestion followed by manual or automated ascorbic acid reduction.	249 256	476, 481 624	384	133	[8] (621)
RADIOLOGICAL						
99. Alpha—Total, pCi per liter.	Proportional or scintillation counter.		648	591 [11]	[24] (75+78)	
100. Alpha—Counting error, pCi per liter.	do		648	594	[24] (79)	
101. Beta—Total, pCi per liter.	Proportional counter.		648	601 [11]	[24] (75+78)	
102. Beta—Counting error, pCi per liter.	do		648	606	[24] (79)	
103. (a) Radium—Total, pCi per liter.	do		661	661		
(b) ^{226}Ra, pCi per liter.	Scintillation counter		667		[24] (81)	
RESIDUE						
104. Total, milligrams per liter	Gravimetric, 103 to 105° C.	270	91			
105. Total dissolved (filterable), milligrams per liter.	Glass fiber filtration, 180° C.	266	92			
106. Total suspended (nonfilterable), milligrams per liter.	Glass fiber filtration, 103 to 105° C.	268	94			
107. Settleable, milliliters per liter or milligrams per liter.	Volumetric or gravimetric		95			
108. Total volatile, milligrams per liter.	Gravimetric, 550° C.	272	95			
109. Specific conductance, micromhos per centimeter at 25° C.	Wheatstone bridge conductimetry.	275	71	120	148	[8] (606)
110. Sulfate (as SO_4), milligrams per liter.	Gravimetric; turbidimetric; or automated colorimetric (barium chloranilate).	277 279	493 496	424 425		[8] (624) [8] (623)
111. Sulfide (as S), milligrams per liter.	Titrimetric—Iodine for levels greater than 1 mg per liter; Methylene blue photometric.	284	505 503		154	
112. Sulfite (as SO_3), milligrams per liter.	Titrimetric, iodine-iodate	285	508	435		
113. Surfactants, milligrams per liter.	Colorimetric (Methylene blue).	157	600	494	[20] (11)	
114. Temperature, degrees C.	Calibrated glass or electrometric thermometer.	286	125		[20] (31)	
115. Turbidity, NTU	Nephelometric	295	132	223	156	

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[1] Recommendations for sampling and preservation of samples according to parameter measured may be found in "Methods for Chemical Analysis of Water and Wastes, 1974" U.S. Environmental Protection Agency, table 2, pp. viii-xii.

[2] All page references for USGS methods, unless otherwise noted, are to Brown, E., Skougstad, M. W., and Fishman, M. J., "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases," U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A1, (1970).

[3] EPA comparable method may be found on indicated page of "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 12th ed. (1975).

[4] Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

[5] The method used must be specified.

[6] The 5 tube MPN is used.

[7] Slack, K. V. and others, "Methods for Collection and Analysis of Aquatic Biological and Mircobiological Samples: U.S. Geological Survey Techniques of Water-Resources Inv. book 5, ch. A4 (1973)."

[8] Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies.

[9] Adequately tested methods for benzidine are not available. Until approved methods are available, the following interim method can be used for the estimation of benzidine: (1) "Method for Benzidine and Its Salts in Wastewaters," available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[10] American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, N.Y. 10018.

[11] Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey for Analysis of Wastewaters," (1976) open-file report 76-177.

[12] Procedures for pentachlorophenol, chlorinated organic compounds ,and pesticides can be obtained from the Environmental Monitoring and Support Lbaoratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[13] Color method (ADMI procedure) available from Environmental Monitoring and Support Lbaoratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[14] For samples suspected of having thiocyanate interference, magnesium chloride is used as the digestion catalyst. In the approved test procedure for cyanides, the recommended catalysts are replaced with 20 ml of a solution of 510 g/l magnesium chloride ($MgCl_2 \cdot 6H_2O$). This substitution will eliminate thiocyanate interference for both total cyanide and cyanide amendable to chlorination measurements.

[15] For the determination of total metals the sample is not filtered before processing. Because vigorous digestion procedures may result in a loss of certain metals through preciptation, a less vigorous treatment is recommended as given on p. 83 (4.1.4) of "Methods for Chemical Analysis of Water and Wastes" (1974). In those instances where a more vigorous digestion is desired the procedure on p. 82 (4.1.3) should be followed. For the measurement of the noble metal series (gold, iridium, osmium, palladium, platimum, rhodium and ruthenium), an aqua regia digestion is to be substituted as follows: Transfer a representitive aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated redistilled HNO_3. Place the beaker on a steam bath and evaporate to dryness. Cool the beaker and cautiously add a 5 ml portion of aqua regia. (Aqua regia is prepared immediately before use by carefully adding 3 volumes of concentrated HCl to one volume of concentrated HNO_3.) Cover the beaker with a watch glass and return to the steam bath. Continue heating the covered beaker for 50 min. Remove cover and evaporate to dryness. Cool and take up the residue in a small quantity of 1:1 HCl. Wash down the beaker walls and watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentration. The sample is now ready for analysis.

[16] As the various furnace devices (flameless AA) are essentially atomic absorption techniques, they are considered to be approved test methods. Methods of standard addition are to be followed as noted in p. 78 of "Methods for Chemical Analysis of Water and Wastes," 1974.

[17] Dissolved metals are defined as those constitutents which will pass though a 0.45 μm membrane filter. A prefiltration is permissible to free the sample from larger suspended solids. Filter the sample as soon as practical after collection using the first 50 to 100 ml to rinse the filter flask. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Discard the portion used to rinse the flask and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled HNO_3 to a pH of 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the samples.

[18] See "Atomic Absorption Newsletter," vol. 13, 75 (1974). Available from Perkin-Elmer Corp., Main Ave., Norwalk, Conn. 06852.

[19] Method available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[20] Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/l and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/l 20 ml of sample should be diluted to 100 ml by adding 40 ml each of 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/l the recommended method is satisfactory.

[21] An automated hydrazine reduction method is available from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[22] A number of such systems manufactured by various companies are considered to be comparable in their performance. In addition, another technique, based on combustion-methane detection is also acceptable.

[23] Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water": U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A3 (1972).

[24] R. F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," "Journal of Chromatography," vol. 47, No. 3, pp. 421-426, 1970.

[25] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only suspended. Therefore, the 2 results must be added together to obtain "total."

[26] Stevens, H. H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation: U.S. Geological Survey Techniques of Water Resources Inv., book 1 (1975)."

4. In § 136.4, the second sentence of paragraph (c) is amended by deleting the word "subchapter" immediately following the phrase "procedure under this" and immediately preceding the word "shall" and replaced with the phrase "paragraph c;" and § 136.4 is amended by adding a new paragraph (d) to read as follows:

§ 136.4 Application for alternate test procedures.

* * * * *

(c) * * * Any application for an alternate test procedure under this paragraph (c) shall: * * *

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. Any application for an alternate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

§ 136.5 [Amended]

5. In § 136.5, paragraph (a) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "test procedure" and before the period that ends the paragraph.

6. In § 136.5, paragraph (b) is amended by inserting in the first sentence the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "such application" and immediately before the comma. The second sentence of paragraph (b) is amended by deleting the phrase "Methods Development and Quality Assurance Research Laboratory" immediately after the phrase "State Permit Program and to the Director of the" at the end of the sentence, and inserting in its place the phrase "Environmental Monitoring and Support Laboratory, Cincinnati."

7. In § 136.5, paragraph (c) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the phrase "application for an alternate test procedure" and immediately before the comma; and by deleting the phrase "Methods Development and Quality Assurance Laboratory" immediately after the phrase "application to the Director of the" and immediately before the phrase "for review and recommendation" and inserting in its place the phrase "Environmental Monitoring and Support Laboratory, Cincinnati."

8. In § 136.5, the first sentence of paragraph (d) is amended by inserting the phrase, "proposed by the responsible person or firm making the discharge," immediately after the phrase, "application for an alternate test procedure," and immediately before the comma.

The second sentence of paragraph (d) is amended by deleting the phrase, "Methods Development and Quality Assurance Research Laboratory," immediately after the phrase, "to the Regional Administrator by the Director of the," and immediately preceding the period ending the sentence and inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."

The third sentence of paragraph (d) is amended by deleting the phrase, "Methods Development and Quality Assurance Research Laboratory," immediately after the phrase, "forwarded to the Director," and immediately before the second comma and by inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."

9. Section 136.5 is amended by the addition of a new paragraph (e) to read as follows:

§ 136.5 Approval of alternate test procedures.

* * * * *

(e) Within ninety days of the receipt by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati of an application for an alternate test procedure for nationwide use, the Director of the Environmental Monitoring and Support Laboratory, Cincinnati shall notify the applicant of his recommendation to the Administrator to approve or reject the application, or shall specify additional information which is required to determine whether to approve the proposed test procedure. After such notification, an alternate method determined by the Administrator to satisfy the applicable requirements of this part shall be approved for nationwide use to satisfy the requirements of this subchapter; alternate test procedures determined by the Administrator not to meet the applicable requirements of this part shall be rejected. Notice of these determinations shall be submitted for publication in the FEDERAL REGISTER not later than 15 days after such notification and determination is made.

[FR Doc.76-35082 Filed 11-30-76;8:45 am]

FEDERAL REGISTER, VOL. 41, NO. 232--WEDNESDAY, DECEMBER 1, 1976, pp. 52780-52786

PART 136--GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Amendment of Regulations; Corrections*

Page	Parameter Number	Correction or Addition
52783	62, Nickel	14th ed. Standard Methods - add "232" to page reference opposite the colorimetric method designation.
52784	89	Parameter and units, change "Nitrate" to "Nitrite"
52784	96, Phenols	Delete the present method designation, "Colorimetric, (4 AAP)," and replace it with the method designation, "Distillation followed by colorimetric, (4 AAP)."
52784	96, Phenols	14th ed. Standard Methods, change "582" to "574"

Amendment; Correction**

52784	106, Total Suspended Residue	In "Method" column, after "103 to 105°C," add: "Glass fiber filtration, 103-105°C, post-washing of residue." Opposite this entry, in the "other Approved Methods" column, add (537).[27] Then, on page 52785, add footnote 27 to read: "27. Standard Methods for the Examination of Water and Wastewater, 13th Edition (1971)."

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*Federal Register, Volume 42, No. 12, Tuesday, January 18, 1977, pp. 3306-3307

**Federal Register, Volume 42, No. 139, Wednesday, July 20, 1977, p. 37205

NPDES/CERTIFICATIONS
ALTERNATIVE TEST PROCEDURES FOR LIMITED USE

TOPIC: Application and approval for individual permit-holders to use alternate test procedures (alternate to those published in the Federal Register for NPDES/certification purposes) for specific discharges.

SOURCE: Federal Register, Title 40, Chapter I, Subchapter D, Part 136:
- Vol 38, No. 199, October 16, 1973
- Vol 41, No. 232, December 1, 1976

A. Application for Alternate Test Procedures

The responsible person or firm making the discharge applies to the EPA Regional Administrator (RA) in the Region where the discharge occurs, through the Director of the State agency having permit-issuing authority. If the state does not issue permits, the application is sent directly to the EPA RA. One must:

1. Provide identifying information, i.e., name, address, permit number, etc.
2. Identify the pollutant or parameter involved.
3. Provide justification for using alternate procedure rather than stipulated test.
4. Provide a detailed description of the proposed procedure with references regarding the applicability to the effluents in question.

B. Approval of Alternate Test Procedures

An EPA Regional Administrator (RA) has the final responsibility for approval.

1. The State Director conducts a technical and administrative review and forwards the application and his recommendation to the RA.
2. The RA conducts a technical and administrative review.
   a. If the State Director recommended rejection for scientific and technical reasons, the RA denies the application and sends a copy of the rejected application and his decision to the applicant, the State Director and to the Director of the Environmental Monitoring and Support Laboratory (EMSL).
   b. Before approving any application, the RA sends a copy of the application to the Director of EMSL for review and recommendation.
3. Prior to 90 days of receipt of the application by the RA, the Director of EMSL forwards to the RA a recommendation providing the scientific and other technical basis for acceptance or rejection of the application.
4. Within 90 days of receipt, the RA notifies the applicant and the appropriate State agency of approval or rejection, or else specifies additional information required for the decision.
5. A copy of all approval and rejection notifications are sent to EMSL for purposes of national coordination.

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NPDES/CERTIFICATIONS
ALTERNATIVE TEST PROCEDURES FOR NATIONWIDE USE

TOPIC: Application and approval by any person, laboratory, manufacturer, etc., for nationwide use of an alternate test procedure (alternate to those published in the Federal Register for NPDES/Certification purposes).

SOURCE: Federal Register, Title 40, Chapter I, Subchapter D, Part 136, Vol 41, No. 232, December 1, 1976

A. Application for Alternate Test Procedures

Any interested person, laboratory, manufacturer, etc., applies to the Director, Environmental Monitoring and Support Laboratory (EMSL), Cincinnati, Ohio 45268. One must:

1. Provide identifying information, i.e., name and address of the responsible person or firm.
2. Identify the pollutant(s) or parameter(s) involved.
3. Provide a detailed description of the proposed procedure with references regarding its applicability.
4. Provide comparability data (proposed procedure compared to procedure published in the Federal Register).

B. Approval of Alternate Test Procedures

The Administrator of the U.S. Environmental Protection Agency has the final responsibility for approval.

1. The Director of EMSL conducts a technical review.
2. Within 90 days of receipt, the Director of EMSL notifies the Administrator of his recommendation to approve or reject the application, or else returns the application for additional information required for the decision.
3. After notification of the EMSL recommendations, the Administrator determines whether or not the alternate test procedures meet the requirements set forth in the Federal Register, i.e., whether the procedures are to be approved or rejected.
4. Within fifteen days of the notification and determination, notice of the final decision is submitted to the Federal Register for publication.

federal register

[6560-01]

ENVIRONMENTAL PROTECTION AGENCY

[FRL 863-7]

APPROVAL OF ALTERNATE WATER POLLUTANT TESTING PROCEDURE

Chemical Oxygen Demand (COD)

232

In accordance with § 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (FEDERAL REGISTER; Vol. 41, No. 233, Wednesday, December 1, 1976, pp. 52780–52786), the Oceanography International Corp. applied for approval of a new test procedure for the measurement of COD. The new procedure incorporates a modification of the digestion technique of the approved procedure in that it uses a semimicro volume of sample and reagents, sealed in a glass ampule, with reflux digestion performed in an oven.

After a through review and evaluation by the Environmental Protection Agency (EPA) of the results of a comparability testing study and other information submitted by the applicant in accordance with § 136.5, the EPA has designated the Oceanography International COD procedure as an approved alternate procedure for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (the EPA's regulation implementing the Freedom of Information Act) at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Oceanography International COD procedure is acceptable for use by any person required to use approved procedures under § 304(g) of the Federal Water Pollution Control Act Amendments of 1972. For such use, the procedure must be used in strict accordance with the methods descriptions. The approved methods descriptions and prepackaged reagent ampules are available for the standard, low value, and high chloride ampule COD procedures from Oceanography International Corp., P.O. Box 2980, 512 West Loop, College Station, Tex. 77840.

Additional information concerning this action may be obtained by writing to the Director, Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

Dated: March 2, 1978.

STEPHEN J. GAGE,
Acting Assistant Administrator for Research and Development.

[FR Doc. 78-5968 Filed 3-6-78; 8:45 am]

VOL. 43, NO. 45—TUESDAY, MARCH 7, 1978

[6560-01]

ENVIRONMENTAL PROTECTION AGENCY

[FRL 956-2]

APPROVAL OF ALTERNATE WATER POLLUTANT TESTING PROCEDURE

Hydrogen Ion (pH) and Ammonia

In accordance with section 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (FEDERAL REGISTER, Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780–52786), the Technicon Instrument Corp. applied for apaproval of new test procedures for the measurement of hydrogen ion (pH) and ammonia. The automated pH electrode procedure is industrial method No. 378-75W/A, October 1976. The automated ammonia electrode procedure is industrial method No. 379-74W/E, February 19, 1976. Manual distillation of samples prior to ammonia measurement may be required as stated in footnote 4, FR 41, No. 232, Wednesday, December 1, 1976, p. 52785.

After a thorough review and evaluation by the Environmental Protection Agency (EPA) of information submitted by the applicant in accordance with § 136.5, the EPA has designated the Technicon methods as approved alternate procedures for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act) at the Environmental Monitoring and Support Laboratory, Cincinnati, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Technicon Instrument Corp. methods are acceptable for use by any person required to use procedures under § 304(h) of the Clean Water Act of 1977. For such use, the procedure must be used in strict accordance with the methods descriptions. The approved methods descriptions are available from Technicon Industrial System, Technicon Instrument Corp., Tarrytown, N.Y. 10591.

Additional information concerning this action may be obtained by writing to the Director, Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

Dated: August 23, 1978.

STEPHAN J. GAGE,
Assistant Administrator for Research and Development.

[FR Doc. 78-24266 Filed 8-28-78; 8:45 am]

FEDERAL REGISTER, VOL. 43, NO. 168—
-TUESDAY, AUGUST 29, 1978

Notice Approval of Alternate Water Pollutant Testing Procedure; Nitrite/Nitrogen

In accordance with § 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (**Federal Register**, Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780–52786), the Hach Chemical Company applied for approval of a new test procedure for the measurement of nitrite/nitrogen. The new Hach procedure is a diazotization method using NitriVer III nitrite reagent.

After a thorough review and evaluation by the Environmental Protection Agency (EPA) of the results of a comparability testing study and other information submitted by the applicant, in accordance with § 136.5, the EPA has designated the Hach procedure as an approved alternate procedure for nationwise use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Hach procedure is acceptable for use by any person required to use approved procedures under § 304(h) of the Clean Water Act Amendments of 1977. For such use, the procedure must be used in strict accordance with the method descriptions. The approved method descriptions and prepackaged reagents are available from the Hach Chemical Company, Post Office Box 389, Loveland, Colorado 80537.

Dated: April 24, 1979.

Stephen J. Gage,

Assistant Administrator for Research and Development.

[FRL 1214-6]

[FR Doc. 79-13321 Filed 4-30-79; 8:45 am]

BILLING CODE 6560-01-M

Federal Register / Vol. 44, No. 85

Tuesday, May 1, 1979

Approval of Alternate Water Pollutant Testing Procedure; Total Manganese

In accordance with § 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (**Federal Register**, Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780–52786), the Hach Chemical Company applied for approval of a new test procedure for the measurement of manganese. The new Hach procedure is a colorimetric procedure using prepackaged reagents based upon the same chemical reactions involved in the approved colorimetric procedure referenced in 40 CFR Part 136.

After a thorough review and evaluation by the Environmental Protection Agency (EPA) of the results of a comparability testing study, and other information submitted by the applicant in accordance with § 136.5, the EPA has designated the Hach procedure as an approved alternate procedure for nationwide use. All information submitted by the applicant is on file and available for public inspection to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Hach procedure is acceptable for use by any person required to use approved procedures under § 304(h) of the Clean Water Act Amendments of 1977. For such use, the procedure must be used in strict accordance with the method descriptions for manganese, periodate oxidation method, "Wastewater Analysis Handbook," Hach Chemical Company, p. 275 and p. 281.

For the analysis of total manganese, addition of acid and digestion as indicated in Footnote 15 of **Federal Register**, Vol. 41, No. 232, Wednesday, December 1, 1976, p. 52785, and § 4.1.4, p. 83 of "Methods for Chemical Analysis of Water and Waste," USEPA, 1974, is the required sample pretreatment. This approved alternate test procedure for total manganese is approved also for the determination of dissolved manganese after sample filtration through a 0.45 micron filter.

The approved method description and prepackaged reagents are available from the Director of Technical Information, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

Dated: June 4, 1979.

Tom Murphy,

Acting Assistant Administrator for Research and Development.

[FR Doc. 79-18[illegible] Filed 6-13-79; 8:45 am]

BILLING CODE 6560-01-M

Federal Register / Vol. 44, No. 116

Thursday, June 14, 1979

[FRL 1470–2]

Approval of Alternate Water Pollutant Testing Procedure; Chemical Oxygen Demand (COD)

In accordance with § 136.5, 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants" (FR Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780–52786), the Hach Chemical Company applied for approval of an alternate test procedure for the measurement of COD. The new procedure incorporates a modification of the digestion technique of the approved procedure in that it uses a semi-micro volume of sample and reagents in a capped vial with reflux digestion performed in an oven.

After a thorough review and evaluation by the U.S. Environment Protection Agency (EPA) of the results of comparability testing studies and other information submitted by the applicant, in accordance with § 136.5, the EPA has designated the Hach procedure as an approved alternate test procedure for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As an approved alternate test procedure, the Hach procedure is acceptable for use by any person required to use approved procedures under § 304(h) of the Clean Water Act Amendments of 1977. For such use, the procedure must be used in strict accordance with the method description, Oxygen Demand, Chemical: Reaction Digestion Method, Water Analysis Handbook, pp. 2–166–2–170, 1979 edition.

The approved method descriptions and prepackaged reagents are available from the Hach Chemical Company, Post Office Box 389, Loveland, Colorado 80537.

Dated: April 14, 1980.

Stephen J. Gage,

Assistant Administrator for Research and Development.

[FR Doc. 80–12172 Filed 4–18–80; 8:45 am]

BILLING CODE 6560–01–M

Federal Register / Vol. 45, No. 78
Monday, April 21, 1980

ENVIRONMENTAL PROTECTION AGENCY

[FRL 1502–2]

Approval of Alternate Water Pollutant Testing Procedures; Total and Dissolved Zinc and Copper

In accordance with § 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants," the Hach Chemical Company applied for approval of alternate test procedures for the measurement of total and dissolved zinc and copper.

After a thorough review and evaluation by the United States Environmental Protection Agency (EPA) of the results of comparability testing studies and other information submitted by the applicant in accordance with § 136.5, the EPA has designated the Hach procedures as approved alternate test procedures for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.

As approved alternate test procedures, the Hach procedures are acceptable for use by any person required to use approved procedures under § 304(h) of the Clean Water Act Amendments of 1977. For such use, the procedures must be used in strict accordance with the method descriptions for copper: Bicinchoninate Method, Method 8506; and for zinc: Zincon Method, Method 8009. In making this approval, the EPA disassociates itself from any statements in the approved Hach Chemical Company procedures which do not specifically pertain to the determination of analytical equivalency.

The approved methods descriptions and prepackaged reagents are available from the Hach Chemical Company, Post Office Box 389, Loveland, Colorado 80537.

Dated: May 21, 1980.

Stephen J. Gage,

Assistant Administrator for Research and Development.

[FR Doc. 80–16224 Filed 5–28–80; 8:45 am]

BILLING CODE 6560–01–M

Federal Register / Vol. 45, No. 105
Thursday, May 29, 1980

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
ENVIRONMENTAL MONITORING AND SUPPORT LABORATORY
CINCINNATI, OHIO 45268

DATE: September 20, 1978

SUBJECT: Use of Chemical Test Kits for Compliance Monitoring

FROM: Dwight G. Ballinger, Director Dwight G Ballinger
Environmental Monitoring and Support
Laboratory - Cincinnati

TO: Regional Quality Assurance Coordinators

A number of Regional Coordinators and Permit staff have received inquiries concerning the use of test kits for the parameters required by the Effluent Guidelines and Compliance Monitoring Sections of PL 92-500. This memorandum defines the position of EMSL on these test methods and contains recommendations for regional and state response to inquiries concerning acceptance.

Our interpretation of Section 304(g), implemented as "Guidelines Establishing Test Procedures for Analysis of Pollutants," in Federal Register, Dec. 1, 1976, is that the test kits are not equivalent to the procedures promulgated in the Federal Register and therefore are subject to the requirements governing alternate procedures. Therefore, it is necessary for the applicant to request the use of these test kits from the appropriate Regional Administrator as presented in the regulations. This recommendation is based upon the following factors:

SAMPLE PREPARATION

Most of the procedures selected for implementation of Section 304(g) are designed to measure the total constituent or element present in the sample or are specifically modified to determine a precisely defined form of the substance. The test kits considered herein generally do not utilize the necessary digestion or pretreatment required to measure the total constituent. In many cases, such as preliminary digestion with acids or distillations, these pretreatments cannot be performed satisfactorily under field conditions. Thus, the final result reported, when a kit is used, is nearly always less than the true value and acceptance of such data will lead to wrong interpretation of effluent loadings.

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INTERFERENCES

It should be recognized that a majority of these test kit measurement techniques were developed for field use on domestic water supplies or relatively clean ambient streams and are not designed to provide for removal of interferences often encountered in municipal and industrial waste effluents.

MEASUREMENT SYSTEMS

In some cases the physical and chemical measurement principles differ from those employed in the reference methods. In addition, proprietary reagents of unknown composition are often provided with little or no information available on the reactions involved. Even though some of the tests are "based on Standard Methods," a number of factors rule out the acceptance of the test procedures as equivalent.

REAGENTS

In most cases the reagent concentrations are not the same as those described in the reference method. Volumes are generally pre-measured under unknown conditions and addition to the sample is often by means of inaccurate droppers or pipets. In short, the quality and quantity of reagents is not under the direct control of the analyst, as required by good analytical technique.

CALIBRATION AND COLOR MEASUREMENT

In most test kits a comparatively simple photometer is provided or available light is used to measure color intensity. Calibration scales are supplied by the manufacturer based upon factors developed under ideal conditions in his laboratory, and recalibration is difficult or not recommended. No provision is made for changes in reagent composition due to inadequate quality control in manufacturing or adverse storage conditions. The photometers usually available will not accept cells of sufficient path length to achieve the required sensitivity, and sealed standards incorporated in the kits are subject to changes in color with time. Finally, the band pass of these photometers is generally too wide for accurate measurement of the appropriate wavelength.

DATA REQUIREMENTS FOR ALTERNATE TEST PROCEDURES

When consideration of the above factors are not sufficient to reject an application for use of test kits, a request should be made for comparative data upon which to judge the applicability of the alternative procedures. The attached protocol is recommended in developing the necessary information. The regulations require that the alternate procedures be used on the waste being monitored and that application be made to the Regional Administrator or State Director having jurisdiction over the permit issuance.

It is recognized that under unusual conditions the applicant may have difficulty obtaining comparative data because of lack of laboratory facilities at remote locations. Even in these cases, the applicant should be urged to obtain adequate data by having the necessary work done at a base laboratory or on contract. The acceptance of alternate procedures without supporting laboratory results may significantly weaken the pollution control efforts intended by PL 92-500.

The use of field test kits, as with other alternate procedures, should be considered on a case-by-case basis, with judgment based on all of the factors involved. No blanket acceptance of test methods will be recommended unless a large volume of data have been accumulated clearly showing that the use of the specific alternate procedure on a wide variety of sample types will provide test results equivalent in precision and accuracy to the reference methods. When such data are available, the method will probably be incorporated in amendments to the listing of Dec. 1, 1976, making further substantiation unnecessary.

Attachment

cc: Walter G. Gilbert, Director
National Training & Operational Technology Center

Attachment

DATA REQUIREMENTS
NATIONWIDE APPROVAL OF ALTERNATE TEST PROCEDURES

1) Five industrial (discharge) sources identified by Standard Industrial Classification (SIC) code or five drinking water sources,

2) Six samples from each source.

3) Four replicate analyses each by the proposed and approved method.

Sources		Samples		Replicates		Methods		Total
5	X	6	X	4	X	2	=	240

DATA REQUIREMENTS
LIMITED - USE APPROVAL OF ALTERNATE TEST PROCEDURE

STATE OR REGIONAL USE

1) Five sources.

2) Three samples from each source.

3) Four replicate analyses each by the proposed and approved method.

Sources		Samples		Replicates		Methods		Total
5	X	3	X	4	X	2	=	120

DATA REQUIREMENTS
LIMITED - USE APPROVAL OF ALTERNATE TEST PROCEDURE

PERMIT HOLDER OR DRINKING WATER SYSTEM

1) Three samples from each source.

2) Four replicate analyses each by the proposed and approved method.

Sources		Samples		Replicates		Methods		Total
1	X	3	X	4	X	2	=	24

STATISTICAL PROTOCOL
APPROVAL OF ALTERNATE TEST PROCEDURES

1) Calculate basic statistics of mean and standard deviation.

2) Test for outliers.

3) Frequency counts and histogram to check distribution.

4) Cochrans test for equality among within-sample standard deviation.

5) F-test for equality of pooled within-sample variances.

6) T-test for equality of method means.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF RESEARCH AND DEVELOPMENT
ENVIRONMENTAL MONITORING AND SUPPORT LABORATORY
CINCINNATI, OHIO 45268

DATE: September 20, 1978

SUBJECT: Use of "Prepared" Reagents in NPDES Compliance

FROM: Dwight G. Ballinger, Director
Environmental Monitoring and Support
Laboratory - Cincinnati

TO: Regional Quality Assurance Coordinators

We encourage the use of pre-prepared solutions and standards by sewage treatment plant operators and others for their required compliance monitoring activities, providing such solutions have been prepared according to the reagents section of the approved methods cited in the Federal Register of December 1, 1976. We would, however, be opposed to the use of commercial reagents that are of unknown composition.

In allowing the use of known pre-prepared solutions and standards, we strongly recommend that the following quality control checks be observed to insure their validity:

1. Date all solutions upon receipt of shipment, store in separate, appropriate area, and observe stated shelf life.
2. Verify that solutions and standards are valid by initially checking them against a quality control check sample available from EMSL through the appropriate Regional QA Coordinator. These check samples are available for most of the common measurements required in the NPDES. They are shipped in a sealed vial as a sample concentrate with the actual composition being provided in a separately sealed envelope.
3. Verify that these solutions are stable on a routine basis by periodically comparing them against a quality control check sample or a standard from another source.

cc: Walter G. Gilbert, Director
National Training & Operational Technology Center

federal register

WEDNESDAY, DECEMBER 24, 1975

THE NATIONAL ARCHIVES OF THE UNITED STATES
1934

PART IV:

ENVIRONMENTAL PROTECTION AGENCY

■

WATER PROGRAMS

National Interim Primary Drinking Water Regulations

Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY

SUBCHAPTER D—WATER PROGRAMS

[FRL 464-7]

PART 141—NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS

On March 14, 1975, the Environmental Protection Agency (EPA) proposed National Interim Primary Drinking Water Regulations pursuant to sections 1412, 1414, 1415, and 1450 of the Public Health Service Act ("the Act"), as amended by the Safe Drinking Water Act ("SDWA," Pub. L. 93-523), 40 FR 11990. EPA held public hearings on the proposed regulations in Boston, Chicago, San Francisco, and Washington during the month of April. Several thousand pages of comments on the proposed regulations were received and evaluated. In addition, the Agency has received comments and information on the proposed regulations from the National Drinking Water Advisory Council, the Secretary of Health, Education, and Welfare, and from numerous others during meetings with representatives of State agencies, public interest groups and others.

The regulations deal only with the basic legal requirements. Descriptive material will be provided in a guidance manual for use by public water systems and the States.

The purpose of this preamble to the final regulations is to summarize the most significant changes made in the proposed regulations as a result of comments received and the further consideration of available information. A more detailed discussion of the comments and of changes in the proposed regulations is attached as Appendix A.

Water Systems Covered

The Safe Drinking Water Act applies to each "public water system," which is defined in Section 1401(4) of the Act as "a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or regularly serves at least twenty-five individuals." Privately owned as well as publicly owned systems are covered. Service "to the public" is interpreted by EPA to include factories and private housing developments. (See generally, House Report, pp. 16–17.)

The definition of "public water system" proposed in the Interim Primary Drinking Water Regulations sought to explain the meaning of the statutory reference to "regular" service. It was proposed to interpret this term as including service for as much as three months during the year. Because the proposed definition would have excluded many large campgrounds, lodges, and other public accommodations which serve large numbers of tourists but which are open for slightly less than three months each year, the definition in the final version covers systems serving an average of at least twenty-five individuals at least 60 days out of the year. The use of a minimum number of days rather than months also makes clear that a system may qualify as a public water system even if it is not open every day during a given month.

Once "public water system" has been defined, it is necessary to define the two major types of public water systems—those serving residents and those serving transients or intermittent users. The possible health effects of a contaminant in drinking water in many cases are quite different for a person drinking the water for a long period of time than for a person drinking the water only briefly or intermittently. Different regulatory considerations may in some cases apply to systems which serve residents as opposed to systems which serve transients or intermittent users. Accordingly, § 141.2(e) makes clear that all "public water systems" fall within either the category of "community water systems" or the category of "non-community water systems." To make clear which regulatory requirements apply to which type of system, the category covered is specifically indicated throughout the regulations.

The proposed regulations defined a "community water system" as "a public water system which serves a population of which 70 percent or greater are residents." Reliance in the proposed definition on the percentage of water system users who are residents would result in treating some fairly large resort communities with many year-round residents as non-community systems. Therefore, the definition of "community water system" has been changed to cover any system which serves at least 15 service connections used by year-round residents or serves at least 25 year-round residents.

Small Community Water Systems

Many community water systems in the country are quite small. Since it is the intention of the Act to provide basically the same level of health protection to residents of small communities as to residents of large cities, and since a number of advanced water treatment techniques are made feasible only by economies of scale, the cost of compliance with the requirements of the Act may pose a serious problem for many small communities. The regulations seek to recognize the financial problems of small communities by requiring more realistic monitoring for systems serving fewer than 1,000 persons. Variances and exemptions authorized by the Act can also assist in dealing with economic problems of small community systems in appropriate cases, at least temporarily. EPA will provide technical assistance on effective treatment techniques which can be used by small systems.

These methods of dealing with the financial problems of some small community systems may not be sufficient in specific instances to make compliance with all applicable regulatory requirements feasible. EPA is commencing a study of potential problems faced by small community systems in meeting applicable requirements under the Act and these regulations, and, if necessary, will make additional adjustments in the Interim Primary Drinking Water Regulations prior to their effective date.

Non-Community Systems

"Non-community systems" are basically those systems which serve transients. They include hotels, motels, restaurants, campgrounds, service stations, and other public accommodations which have their own water system and which have at least 15 service connections or serve water to a daily average of at least 25 persons. Some schools, factories and churches are also included in this category. It is conservatively estimated that there are over 200,000 non-community water systems in the country. However, it should be recognized that while their number is large, they normally are not the principal source of water for the people they serve.

The regulations as proposed would have applied all maximum contaminant levels to non-community systems as well as to community systems. This approach failed to take into account the fact that the proposed maximum contaminant levels for organic chemicals and most inorganic chemicals were based on the potential health effects of long-term exposure. Those levels are not necessary to protect transients or intermittent users. Therefore, the final regulations provide that maximum contaminant levels for organic chemicals, and for inorganic chemicals other than nitrates, are not applicable to non-community systems. An exception was made for nitrates because they can have an adverse health effect on susceptible infants in a short period of time.

Even without monitoring for organic chemicals or most inorganic chemicals, in the initial stages of implementation of the drinking water regulations, monitoring results from tens of thousands of non-community systems could overwhelm laboratory capabilities and other resources. This could delay effective implementation of the regulations with respect to the community systems which provide the water which Americans drink every day. To avoid this result, non-community systems will be given two years after the effective date of the regulations to commence monitoring. In the meantime, non-community systems which already monitor their water are encouraged to continue to do so, and the States are encouraged to take appropriate measures to test or require monitoring for non-community systems that serve large numbers of people.

Of course, non-community systems which pose a threat to health should be dealt with as quickly as possible. The maximum contaminant levels applicable to non-community water systems therefore will take effect 18 months after promulgation, at the same time as levels applicable to community systems. Inspection and enforcement authority will apply to non-community systems at the same time as to community systems.

Sanitary Surveys

EPA encourages the States to conduct sanitary surveys on a systematic basis.

These on-site inspections of water systems are more effective in assuring safe water to the public than individual tests taken in the absence of sanitary surveys. The regulations provide that monitoring frequencies for coliform bacteria can be changed by the entity with primary enforcement responsibility for an individual non-community system, and in certain circumstances for an individual community system, based on the results of a sanitary survey.

MAXIMUM CONTAMINANT LEVELS

Numerous comments were received by EPA on the substances selected for the establishment of maximum contaminant levels and on the levels chosen. Congress anticipated that the initial Interim Primary Drinking Water Regulations would be based on the Public Health Service Standards of 1962, and this Congressional intent has been followed. Comments received on the various levels did not contain new data sufficient to require the establishment of levels different from those contained in the Public Health Service Standards.

WATER CONSUMPTION

The maximum contaminant levels are based, directly or indirectly, on an assumed consumption of two liters of water per day. The same assumption was used in the 1962 Standards. This assumption has been challenged because of instances where much higher water consumption rates occur. EPA's justification for using the two-liter figure is that it already represents an above average water or water-based fluid intake. Moreover, while the factor of safety may be somewhat reduced when greater quantities of water are ingested, the maximum contaminant levels based on the two-liter figure provide substantial protection to virtually all consumers. If, as has been suggested, a water consumption rate of eight liters per day is used as the basis for maximum contaminant level, all of the proposed MCL's would have to be divided by four, greatly increasing the monitoring difficulties, and in some cases challenging the sensitivity of accepted analytical procedures. It could be expected, in such a case, that the maximum contaminant levels would be exceeded to a significant degree, and that specialized treatment techniques would be required to order that the contaminant levels would be reduced. The economic impact of a move in this direction would be enormous. It is not technically or economically feasible to base maximum contaminant levels on unusually high consumption rates.

SAFETY FACTORS

A question was raised about the fact that different safety factors are contained in various maximum contaminant levels. The levels are not intended to have a uniform safety factor, at least partly because the knowledge of and the nature of the health risks of the various contaminants vary widely. The levels set are the result of experience, evaluation of the available data, and professional judgment. They have withstood the test of time and of professional review. They are being subjected to further review by the National Academy of Sciences in connection with development of data for the Revised Primary Drinking Water Regulations.

MCL'S BASED ON TEMPERATURE

A question was also raised as to whether ranges of maximum contaminant levels should be established on the basis of the climate in the area served by the public water system, as was done with fluoride. EPA believes that the use of a temperature scale for fluoride is more appropriate than for other chemicals because of the studies available on the fluoride-temperature relationship and because there is a small margin with fluoride between beneficial levels and levels that cause adverse health effects.

MCL'S DELETED

Three proposed maximum contaminant levels have been eliminated in the final regulations because they are not justified by the available data. One of these is carbon chloroform extract (CCE), which is discussed separately below. The others are the proposed levels for the standard bacterial plate count and cyanide. In the case of the plate count, it is believed that the coliform limits contained in the regulations, combined with the turbidity maximum contaminant level, adequately deal with bacterial contamination. However, EPA continues to believe that the standard plate count is a valid indicator of bacteriological quality of drinking water, and recommends that it be used in appropriate cases in conjunction with the coliform tests as an operational tool.

The proposed maximum contaminant level for cyanide was eliminated because the possibility of cyanide contamination can be effectively addressed only by the use of emergency action, such as under Section 1431 of the Act. EPA's 1969 Community Water Supply Study did not reveal a single instance in which cyanide was present in a water system at a level greater than one-thousandth of the level at which cyanide is toxic to humans.

Available data indicate that cyanide will be present in water systems at toxic levels only in the event of an accident, such as a spill from a barge collision. Maximum contaminant levels are not the appropriate vehicle for dealing with such rare, accidental contamination.

Heptachor, heptachlor epoxide and chlordane have also been removed from the list of maximum contaminant levels at least temporarily in view of the pending cancellation and suspension proceedings under the Federal Insecticide, Fungicide and Rodenticide Act involving those pesticides. When the results of these proceedings are available, EPA will again consider whether maximum contaminant levels should be established for those three pesticides.

SODIUM AND SULFATES

A number of comments were received on the potential health effects of sodium and sulfates. The National Drinking Water Advisory Council has recommended that consideration be given to the monitoring of these constituents but has not recommended the adoption of maximum contaminant levels because available data do not support the adoption of any specific levels. EPA has requested the National Academy of Sciences to include sodium and sulfates among the contaminants to be studied by NAS, and to include information on the health effects of sodium and sulfates in the report to be made by NAS in December 1976.

Since a number of persons suffer from diseases which are influenced by dietary sodium intake and since there are others who wish to restrict their sodium intake, it is desirable that the sodium content of drinking water be known. Those affected can, by knowing the sodium concentration in their drinking water, make adjustments to their diets or, in extreme cases, seek alternative sources of water to be used for drinking and food preparation. It is recommended that the States institute programs for regular monitoring of the sodium content of drinking water served to the public, and for informing physicians and consumers of the sodium concentration in drinking water.

A relatively high concentration of sulfate in drinking water has little or no known laxative effect on regular users of the water, but transcients using such water sometimes experience a laxative effect. It is recommended that the States institute monitoring programs for sulfates, and that transients be notified if the sulfate content of the water is high. Such notification should include an assessment of the possible physiological effects of consumption of the water.

PCB'S AND ASBESTOS

An interagency comment expressed concern for asbestos and PCB's in the environment and noted the need for at least a monitoring requirement, if not for MCL's, for these contaminants. EPA is also concerned, but for the moment lacks sufficient evidence regarding analytical methods, health effects, or occurrence in the environment to establish MCL's. The Agency is conducting research and cooperating in research projects to develop criteria for establishing needed limits as quickly as possible. A monitoring study on a number of organic chemical contaminants, including PCB's for which MCL's are not being established at this time, will be contained in an organic chemical monitoring regulation that is being promulgated with these regulations. Regarding asbestos, HEW and EPA are sponsoring a number of studies this year at an approximate cost of $16 million to establish health effects, anaylitical methods and occurrence.

POINT OF MEASUREMENT

Other comments on maximum contaminant levels focused on the proposed requirement that such levels be tested at the consumer's tap. Concern was expressed over the inability of the public water system to control potential sources of contaminants which are under the control of the consumer.

The promulgated definition of "maximum contaminant level," § 141.2(d), retains the requirement that the maximum contaminant level be measured at the tap except in the case of turbidity, which should be measured at the point of entry to the distribution system. However, the definition has been expanded to make clear that contaminants added to the water by circumstances under the control of the consumer are not the responsibility of the supplier of water, unless the contaminants result from corrosion of piping and plumbing resulting from the quality of the water supplied. It should be noted, however, that this requirement should not be interpreted as to discourage local, aggressive cross connection control measures.

COLIFORM BACTERIA MCL'S

The promulgated MCL's for coliform bacteria are basically the 1962 Public Health Service Standards, with minor refinements and clarifications. However, further changes may be desirable. For example, the MCL's for the membrane filter analytical method do not resolve the question of how many coliform bacteria are assumed to be present in a single highly contaminated sample. Some laboratories assume an upper limit of 50, while others seek to continue to count individual bacteria to a level of 100 or even higher in a single sample. The upper limit assumed will affect the monthly average which is calculated to determine compliance with the MCL's.

Another question relating to the coliform bacteria MCL's is the matter of possible spurious positive samples. As the regulations are written, all routine samples taken to determine compliance with the MCL's must be counted, regardless of the results of analysis of any check samples that may be taken. The reason for this is that bacterial contamination is often intermittent or transient, and as a result negative check samples taken a day or more after a positive sample cannot demonstrate that the positive result was in error. It may be possible, however, to prescribe a means of dealing with spurious positive results without compromising the integrity of the MCL's.

A third question concerning the MCL's for coliform bacteria is the relationship of monthly averages of coliform bacteria levels to monthly percentages of positive samples. For example, the monthly average MCL for the membrane filter method is violated if the monthly average exceeds one coliform bacterium per sample. However, for purposes of determining whether the monthly-percentage-of-positive-samples MCL is violated, a sample is counted as positive only if it contains more than four coliform bacteria. Thus, it is possible, particularly when a relatively small number of samples is taken, for a system to fail the monthly average MCL even when no single sample taken during the month is out of compliance with the limit.

These and other questions concerning the coliform bacteria MCL's will be reviewed further by EPA. If review indicates that changes in the MCL's are desirable, those changes will be made as soon as possible but within 6 months, in time to take effect at the same time as the initial Interim Primary Drinking Water Regulations.

ORGANIC CHEMICALS

The proposed maximum contaminant levels for organic pesticides, other than the three which are the subject of cancellation and suspension proceedings, have been retained. It is anticipated that additional organic pesticides will be added to the regulations if surveys of pesticides in drinking water being conducted by EPA indicate that this is needed.

The proposed regulations also contained a maximum contaminant level for organic chemicals obtained by the carbon chloroform extract (CCE) method. It was anticipated by Congress that organic chemicals would be dealt with primarily in the Revised Primary Drinking Water Regulations because of the paucity of accurate data on the health effects of various organic chemicals, the large number of such chemicals, uncertainities over appropriate treatment techniques, and the need for additional information on the incidence of specific organic chemicals in drinking water supplies. EPA thought that the CCE standard might provide an appropriate means of dealing with organic chemicals as a class pending action on the Revised Primary Regulations.

The CCE standard was originally developed as a test for undesirable tastes and odors in drinking water. As concern developed over the health effects of organic chemicals, the possibility of using CCE as a health standard rather than an esthetic standard was considered.

As pointed out by numerous comments, CCE has many failings as an indicator of health effects of organic chemicals. To begin with, the test obtains information on only a fraction of the total amount of organic chemicals in the water sampled. Furthermore, there is serious question as to the reliability of CCE in identifying those organic chemicals which are most suspected of adverse health effects. In addition, there are no existing data on which a specific level for CCE can be established on a rational basis. To establish a maximum contaminant level under these circumstances would almost certainly do more harm than good. It could give a false sense of security to persons served by systems which are within the established level and a false sense of alarm to persons served by systems which exceed the level. It also would divert resources from efforts to find more effective ways of dealing with the organic chemicals problem.

EPA believes that the intelligent approach to the organic chemicals question is to move ahead as rapidly as possible along two fronts. First, EPA is adopting simultaneously with these regulations a Subpart E of Part 141, containing requirements for organic chemical monitoring pursuant to Sections 1445 and 1450 of the Act.

The regulations require that designated public water systems collect samples of raw and treated water for submission to EPA for organics analysis. EPA will analyze the samples for a number of broad organic parameters, including carbon chloroform extract (CCE), volatile and non-volatile total organic carbon (VTOC and NVTOC), total organic chlorine (TOCl), ultraviolet absorbancy, and fluorescence. In addition, monitoring will be required for probably 21 specific organic compounds. Selection of the specific compounds has been based on the occurrence or likelihood of occurrence in treated water, toxicity data and availability of practical analytical methods. Laboratory analyses will be used to evaluate the extent and nature of organic chemical contamination of drinking water, to evaluate the validity of the general organic parameters as surrogates for measures of harmful organic chemicals, and to determine whether there is an adequate basis for establishing maximum contaminant levels for specific organics or groups of organics.

Second, EPA is embarking on an intensive research program to find answers to the following four questions:

1. What are the effects of commonly occurring organic compounds on human health?

2. What analytical procedures should be used to monitor finished drinking water to assure that any Primary Drinking Water Regulations dealing with organics are met?

3. Because some of these organic compounds are formed during water treatment, what changes in treatment practices are required to minimize the formation of these compounds in treated water?

4. What treatment technology must be applied to reduce contaminant levels to concentrations that may be specified in the Primary Drinking Water Regulations?

This research will involve health-effects and epidemiological studies, investigations of analytical methodology, and pilot plant and field studies of organic removal unit processes. Some phases of the research are to be completed by the end of this year, while much of the remainder are to be completed within the next calendar year.

As soon as sufficient information is derived from the monitoring program and related research, the Interim Primary Drinking Water Regulations will be amended so that the organic chemicals problem can be dealt with without delay. The monitoring process will be completed within 1 year.

During the interim period, while satisfactory MCL's for organic contamination in drinking water are being developed, EPA will act in specific cases where appropriate to deal with organic contamination. If the EPA monitoring program reveals serious specific cases of contamination, EPA will work with State and local authorities to identify the source and nature of the problem and to take remedial action. EPA will also aid the States in identifying additional community water supplies that require analysis.

PUBLIC NOTICE

The public notice requirements proposed in § 141.32 did not distinguish between community and non-community public water systems. They would have required that public notice of non-compliance with applicable regulations be made by newspaper, in water bills, and by other media for all public water systems. These requirements are inappropriate and ineffective in the case of most non-community water systems. Those systems principally serve transients who do not receive water bills from the system and who probably are not exposed significantly to the local media. A more effective approach would be to require notice that can inform the transient before he drinks the system's water, and thereby both warn the transient and provide an incentive to the supplier of water to remedy the violation. Accordingly, Section 141.32 as adopted provides that in the case of non-community systems, the entity with primary enforcement responsibility shall require that notice be given in a form and manner that will insure that the public using the public water system is adequately informed.

The proposed public notice requirements also failed to distinguish between different types of violations of the Interim Primary Drinking Water Regulations. Since the urgency and importance of a notice varies according to the nature of the violation involved, § 141.32 as promulgated seeks to match the type of notice required with the type of violation involved. Written notice accompanying a water bill or other direct notice by mail is required for all violations of the regulations, including violations of monitoring requirements, and for the grant of a variance or exemption. In addition, notice by newspaper and notification to radio and television stations is required whenever a maximum contaminant level is exceeded, or when the entity with primary enforcement responsibility requires such broader notice.

QUALITY CONTROL AND TESTING PROCEDURES

Section 1401(1) of the Act defines "primary drinking water regulation" to include "quality control and testing procedures." The promulgated regulations include testing requirements for each maximum contaminant level, including check samples and special samples in appropriate cases. The regulations also specify the procedures to be followed in analyzing samples for each of the maximum contaminant levels. These procedures will be updated from time to time as advances are made in analytical methods. For example, references to "Standard Methods for the Examination of Water and Wastewater" are to the current, 13th, edition, but these references will be changed to cite the 14th edition when it is available in the near future.

A key element of quality control for public water systems is accurate laboratory analysis. Section 141.28 of the regulations provides that analyses conducted for the purpose of determining compliance with maximum contaminant levels must be conducted by a laboratory approved by the entity with primary enforcement responsibility. EPA will develop as soon as possible, in cooperation with the States and other interested parties, criteria and procedures for laboratory certification. A State with primary enforcement responsibility will have a laboratory certified by EPA pursuant to the prescribed criteria and procedures, and in turn will certify laboratories within the State.

Record-keeping requirements and reports to the State also will assist in quality control efforts.

RECORD-KEEPING

Adequate record-keeping is necessary for the proper operation and administration of a public water system. It is also important for providing information to the public, providing appropriate data for inspection and enforcement activities and providing information on which future regulations can be based. Accordingly, a new § 141.33 has been added to the regulations to require that each public water system maintain records of sample analyses and of actions to correct violations of the Primary Drinking Water Regulations.

ECONOMIC AND COST ANALYSIS

A comprehensive economics study has been made of the Interim Primary Drinking Water Regulations. This study estimates the costs of the regulations, evaluates the potential economic impact, and considers possible material and labor shortages. The results of this analysis are summarized here.

Total investment costs to community water systems to achieve compliance with these regulations are estimated to be between $1,050 and $1,765 million. It is estimated that non-community systems will invest an additional $24 million. The range of the estimate is due to uncertainty as to the design flow that will be used in installing treatment facilities. Systems not in compliance will have to consider sizing their new components to reflect average daily flow conditions, or maximum daily flow conditions in cases where system storage is not adequate.

This investment will be spread over several years. Investor-owned systems will bear about one-fourth of these costs, and publicly-owned systems the remainder. It is not anticipated that systems will have difficulty financing these capital requirements.

In annual terms, national costs are expected to be within the following ranges:

	In millions
Capital costs	$146-247
Operations and maintenance	263-263
Monitoring (routine only)	17- 35
Total	$426-545

Although these aggregate figures are large, most water consumers will not be significantly affected. For those users in systems serving 10,000 persons or more, the average annual treatment cost per capita may increase from less than $1.00 for systems requiring disinfection and lead control, to between $15 to $35 for control of turbidity and heavy metal removal. For systems serving less than 100 persons, the average annual per capita costs of disinfection, lead control and fluoride/arsenic removal are estimated to be between $2.10 and $11.80. However, if turbidity control or heavy metal removal were required in a system of this size then costs are expected to range from $52 to $237 per year per capita. EPA is aware of the serious potential economic impact on users in these small systems. However, the legislative history specifies that the regulations should be based on costs that can be reasonably afforded by large metropolitan or regional systems. Further economic evaluation of these systems is being conducted, and realistic options for these small systems are being reviewed. Options that will be under consideration include less costly treatment technologies; formation of regional systems; and use of alternative water sources. Industrial and commercial users, whether providing their own water or using public systems, are not expected to be significantly affected by these regulations.

Possible constraints to the implementation of the interim primary regulations were examined. Although there will be an increase in demand for chemicals, manpower, laboratories, and construction of treatment facilities, it is not anticipated that any of these factors will be a serious obstacle to implementation of these regulations over a reasonable time frame.

For the reasons given above, Chapter 40 of the Code of Federal Regulations is hereby amended by the addition of the following new Part 141. These regulations will take effect 18 months after promulgation.

(It is hereby certified that the economic and inflationary impacts of these regulations have been carefully evaluated in accordance with Executive Order 11821)

Dated: December 10, 1975.

RUSSELL E. TRAIN,
Administrator.

Subpart A—General

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Subpart D—Reporting, Public Notification, and Record keeping

141.31 Reporting requirements.
141.32 Public notification of variances, exemptions, and non-compliance with regulations.
141.33 Record maintenance.

AUTHORITY: Secs. 1412, 1414, 1445, and 1450 of the Public Health Service Act, 88 Stat. 1660 (42 U.S.C. 300g-1, 300g-3, 300j-4, and 300j-9).

Subpart A—General

§ 141.1 Applicability.

This part establishes primary drinking water regulations pursuant to section 1412 of the Public Health Service Act, as amended by the Safe Drinking Water Act (Pub. L. 93-523); and related regulations applicable to public water systems.

§ 141.2 Definitions.

As used in this part, the term:

(a) "Act" means the Public Health Service Act, as amended by the Safe Drinking Water Act, Pub. L. 93-523.

(b) "Contaminant" means any physical, chemical, biological, or radiological substance or matter in water.

(c) "Maximum contaminant level" means the maximum permissible level of a contaminant in water which is delivered to the free flowing outlet of the ultimate user of a public water system, except in the case of turbidity where the maximum permissible level is measured at the point of entry to the distribution system. Contaminants added to the water under circumstances controlled by the user, except those resulting from corrosion of piping and plumbing caused by water quality, are excluded from this definition.

(d) "Person" means an individual, corporation, company, association, partnership, State, municipality, or Federal agency.

(e) "Public water system" means a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system. A public water system is either a "community water system" or a "non-community water system."

(i) "Community water system" means a public water system which serves at least 15 service connections used by year-round residents or regularly serves at least 25 year-round residents.

(ii) "Non-community water system" means a public water system that is not a community water system.

(f) "Sanitary survey" means an on-site review of the water source, facilities, equipment, operation and maintenance of a public water system for the purpose of evaluating the adequacy of such source, facilities, equipment, operation and maintenance for producing and distributing safe drinking water.

(g) "Standard sample" means the aliquot of finished drinking water that is examined for the presence of coliform bacteria.

(h) "State" means the agency of the State government which has jurisdiction over public water systems. During any period when a State does not have primary enforcement responsibility pursuant to Section 1413 of the Act, the term "State" means the Regional Administrator, U.S. Environmental Protection Agency.

(i) "Supplier of water" means any person who owns or operates a public water system.

§ 141.3 Coverage.

This part shall apply to each public water system, unless the public water system meets all of the following conditions:

(a) Consists only of distribution and storage facilities (and does not have any collection and treatment facilities);

(b) Obtains all of its water from, but is not owned or operated by, a public water system to which such regulations apply;

(c) Does not sell water to any person; and

(d) Is not a carrier which conveys passengers in interstate commerce.

§ 141.4 Variances and exemptions.

Variances or exemptions from certain provisions of these regulations may be granted pursuant to Sections 1415 and 1416 of the Act by the entity with primary enforcement responsibility. Provisions under Part 142, *National Interim Primary Drinking Water Regulations Implementation*—subpart E (Variances) and subpart F (Exemptions)—apply where EPA has primary enforcement responsibility.

§ 141.5 Siting requirements.

Before a person may enter into a financial commitment for or initiate construction of a new public water system or increase the capacity of an existing public water system, he shall notify the State and, to the extent practicable, avoid locating part or all of the new or expanded facility at a site which:

(a) Is subject to a significant risk from earthquakes, floods, fires or other disasters which could cause a breakdown of the public water system or a portion thereof; or

(b) Except for intake structures, is within the floodplain of a 100-year flood or is lower than any recorded high tide where appropriate records exist.

The U.S. Environmental Protection Agency will not seek to override land use decisions affecting public water systems siting which are made at the State or local government levels.

§ 141.6 Effective date.

The regulations set forth in this part shall take effect 18 months after the date of promulgation.

Subpart B—Maximum Contaminant Levels

§ 141.11 Maximum contaminant levels for inorganic chemicals.

(a) The maximum contaminant level for nitrate is applicable to both community water systems and non-community water systems. The levels for the other inorganic chemicals apply only to community water systems. Compliance with maximum contaminant levels for inorganic chemicals is calculated pursuant to § 141.23.

(b) The following are the maximum contaminant levels for inorganic chemicals other than fluoride:

Contaminant	*Level, milligrams per liter*
Arsenic	0.05
Barium	1.
Cadmium	0.010
Chromium	0.05
Lead	0.05
Mercury	0.002
Nitrate (as N)	10.
Selenium	0.01
Silver	0.05

(c) When the annual average of the maximum daily air temperatures for the location in which the community water system is situated is the following, the maximum contaminant levels for fluoride are:

Temperature Degrees Fahrenheit	Degrees Celsius	Level, milligrams per liter
53.7 and below	12.0 and below	2.4
53.8 to 58.3	12.1 to 14.6	2.2
58.4 to 63.8	14.7 to 17.6	2.0
63.9 to 70.6	17.7 to 21.4	1.8
70.7 to 79.2	21.5 to 26.2	1.6
79.3 to 90.5	26.3 to 32.5	1.4

§ 141.12 Maximum contaminant levels for organic chemicals.

The following are the maximum contaminant levels for organic chemicals. They apply only to community water systems. Compliance with maximum contaminant levels for organic chemicals is calculated pursuant to § 141.24.

	Level, milligrams per liter
(a) Chlorinated hydrocarbons:	
Endrin (1,2,3,4,10, 10-hexachloro-6,7-epoxy-1,4, 4a,5,6,7,8,8a-octahydro-1,4-endo, endo-5,8 - dimethano naphthalene).	0.0002
Lindane (1,2,3,4,5,6-hexachlorocyclohexane, gamma isomer).	0.004
Methoxychlor (1,1,1-Trichloro-2, 2 - bis [p-methoxyphenyl] ethane).	0.1
Toxaphene ($C_{10}H_{10}Cl_8$-Technical chlorinated camphene, 67-69 percent chlorine).	0.005

(b) Chlorophenoxys:	
2,4 - D, (2,4-Dichlorophenoxyacetic acid).	0.1
2,4,5-TP Silvex (2,4,5-Trichlorophenoxypropionic acid).	0.01

§ 141.13 Maximum contaminant levels for turbidity.

The maximum contaminant levels for turbidity are applicable to both community water systems and non-community water systems using surface water sources in whole or in part. The maximum contaminant levels for turbidity in drinking water, measured at a representative entry point(s) to the distribution system, are:

(a) One turbidity unit (TU), as determined by a monthly average pursuant to § 141.22, except that five or fewer turbidity units may be allowed if the supplier of water can demonstrate to the State that the higher turbidity does not do any of the following:

(1) Interfere with disinfection;

(2) Prevent maintenance of an effective disinfectant agent throughout the distribution system; or

(3) Interfere with microbiological determinations.

(b) Five turbidity units based on an average for two consecutive days pursuant to § 141.22.

§ 141.14 Maximum microbiological contaminant levels.

The maximum contaminant levels for coliform bacteria, applicable to community water systems and non-community water systems, are as follows:

(a) When the membrane filter technique pursuant to § 141.21(a) is used, the number of coliform bacteria shall not exceed any of the following:

(1) One per 100 milliliters as the arithmetic mean of all samples examined per month pursuant to § 141.21 (b) or (c);

(2) Four per 100 milliliters in more than one sample when less than 20 are examined per month; or

(3) Four per 100 milliliters in more than five percent of the samples when 20 or more are examined per month.

(b)(1) When the fermentation tube method and 10 milliliter standard portions pursuant to § 141.21(a) are used, coliform bacteria shall not be present in any of the following:

(i) more than 10 percent of the portions in any month pursuant to § 141.21 (b) or (c);

(ii) three or more portions in more than one sample when less than 20 samples are examined per month; or

(iii) three or more portions in more than five percent of the samples when 20 or more samples are examined per month.

(2) When the fermentation tube method and 100 milliliter standard portions pursuant to § 141.21(a) are used, coliform bacteria shall not be present in any of the following:

(i) more than 60 percent of the portions in any month pursuant to § 141.21 (b) or (c);

(ii) five portions in more than one sample when less than five samples are examined per month; or

(iii) five portions in more than 20 percent of the samples when five or more samples are examined per month.

(c) For community or non-community systems that are required to sample at a rate of less than 4 per month, compliance with paragraphs (a), (b)(1), or (b)(2) of this section shall be based upon sampling during a 3 month period, except that, at the discretion of the State, compliance may be based upon sampling during a one-month period.

Subpart C—Monitoring and Analytical Requirements

§ 141.21 Microbiological contaminant sampling and analytical requirements.

(a) Suppliers of water for community water systems and non-community water systems shall analyze for coliform bacteria for the purpose of determining compliance with § 141.14. Analyses shall be conducted in accordance with the analytical recommendations set forth in "Standard Methods for the Examination of Water and Wastewater," American Public Health Association, 13th Edition, pp. 662–688, except that a standard sample size shall be employed. The standard sample used in the membrane filter procedure shall be 100 milliliters. The standard sample used in the 5 tube most probable number (MPN) procedure (fermentation tube method) shall be 5 times the standard portion. The standard portion is either 10 milliliters or 100 milliliters as described in § 141.14 (b) and (c). The samples shall be taken at points which are representative of the conditions within the distribution system.

(b) The supplier of water for a community water system shall take coliform density samples at regular time intervals, and in number proportionate to the population served by the system. In no event shall the frequency be less than as set forth below:

Population served:	*Minimum number of samples per month*
25 to 1,000	1
1,001 to 2,500	2
2,501 to 3,300	3
3,301 to 4,100	4
4,101 to 4,900	5
4,901 to 5,800	6
5,801 to 6,700	7
6,701 to 7,600	8
7,601 to 8,500	9
8,501 to 9,400	10
9,401 to 10,300	11
10,301 to 11,100	12
11,101 to 12,000	13
12,001 to 12,900	14
12,901 to 13,700	15
13,701 to 14,600	16
14,601 to 15,500	17
15,501 to 16,300	18
16,301 to 17,200	19
17,201 to 18,100	20
18,101 to 18,900	21
18,901 to 19,800	22
19,801 to 20,700	23
20,701 to 21,500	24
21,501 to 22,300	25
22,201 to 23,200	26
23,201 to 24,000	27
24,001 to 24,900	28
24,901 to 25,000	29
25,001 to 28,000	30
28,001 to 33,000	35
33,001 to 37,000	40
37,001 to 41,000	45
41,001 to 46,000	50
46,001 to 50,000	55
50,001 to 54,000	60
54,001 to 59,000	65
59,001 to 64,000	70
64,001 to 70,000	75
70,001 to 76,000	80
76,001 to 83,000	85
83,001 to 90,000	90
90,001 to 96,000	95
96,001 to 111,000	100
111,001 to 130,000	110
130,001 to 160,000	120
160,001 to 190,000	130
190,001 to 220,000	140
220,001 to 250,000	150
250,001 to 290,000	160
290,001 to 320,000	170
320,001 to 360,000	180
360,001 to 410,000	190
410,001 to 450,000	200
450,001 to 500,000	210
500,001 to 550,000	220
550,001 to 600,000	230
600,001 to 660,000	240
660,001 to 720,000	250
720,001 to 780,000	260
780,001 to 840,000	270
840,001 to 910,000	280
910,001 to 970,000	290
970,001 to 1,050,000	300
1,050,001 to 1,140,000	310
1,140,001 to 1,230,000	320
1,230,001 to 1,320,000	330
1,320,001 to 1,420,000	340
1,420,001 to 1,520,000	350
1,520,001 to 1,630,000	360
1,630,001 to 1,730,000	370
1,730,001 to 1,850,000	380
1,850,001 to 1,970,000	390
1,970,001 to 2,060,000	400
2,060,001 to 2,270,000	410
2,270,001 to 2,510,000	420
2,510,001 to 2,750,000	430
2,750,001 to 3,020,000	440
3,020,001 to 3,320,000	450
3,320,001 to 3,620,000	460
3,620,001 to 3,960,000	470
3,960,001 to 4,310,000	480
4,310,001 to 4,690,000	490
4,690,001 or more	500

Based on a history of no coliform bacterial contamination and on a sanitary survey by the State showing the water system to be supplied solely by a protected ground water source and free of sanitary defects, a community water system serving 25 to 1,000 persons, with written permission from the State, may reduce this sampling frequency except that in no case shall it be reduced to less than one per quarter.

(c) The supplier of water for a non-community water system shall sample for coliform bacteria in each calendar quarter during which the system provides water to the public. Such sampling shall begin within two years after the effective date of this part. If the State, on the basis of a sanitary survey, determines that some other frequency is more appropriate, that frequency shall be the frequency required under these regulations. Such frequency shall be confirmed or changed on the basis of subsequent surveys.

(d)(1) When the coliform bacteria in a single sample exceed four per 100 milliliters (§ 141.14(a)), at least two consecutive daily check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily, or at a frequency established by the State, until the results obtained from at least two consecutive check samples show less than one coliform bacterium per 100 milliliters.

(2) When coliform bacteria occur in three or more 10 ml portions of a single sample (§ 141.14(b)(1)), at least two consecutive daily check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily, or at a frequency established by the State, until the results obtained from at least two consecutive check samples show no positive tubes.

(3) When coliform bacteria occur in all five of the 100 ml portions of a single sample (§ 141.14(b)(2)), at least two daily check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily, or at a frequency established by the State, until the results obtained from at least two consecutive check samples show no positive tubes.

(4) The location at which the check samples were taken pursuant to paragraphs (d) (1), (2), or (3) of this section shall not be eliminated from future sampling without approval of the State. The results from all coliform bacterial analyses performed pursuant to this subpart, except those obtained from check samples and special purpose samples, shall be used to determine compliance with the maximum contaminant level for coliform bacteria as established in § 141.14. Check samples shall not be included in calculating the total number of samples taken each month to determine compliance with § 141.21 (b) or (c).

(e) When the presence of coliform bacteria in water taken from a particular sampling point has been confirmed by any check samples examined as directed in paragraphs (d) (1), (2), or (3) of this section, the supplier of water shall report to the State within 48 hours.

(f) When a maximum contaminant level set forth in paragraphs (a), (b) or (c) of § 141.14 is exceeded, the supplier of water shall report to the State and notify the public as prescribed in § 141.31 and § 141.32.

(g) Special purpose samples, such as those taken to determine whether disinfection practices following pipe placement, replacement, or repair have been sufficient, shall not be used to determine compliance with § 141.14 or § 141.21 (b) or (c).

(h) A supplier of water of a community water system or a non-community water system may, with the approval of the State and based upon a sanitary survey, substitute the use of chlorine residual monitoring for not more than 75 percent of the samples required to be taken by paragraph (b) of this section, *Provided*, That the supplier of water takes chlorine residual samples at points which are representative of the conditions within the distribution system at the frequency of at least four for each substituted microbiological sample. There shall be at least daily determinations of chlorine residual. When the supplier of water exercises the option provided in this paragraph (h) of this section, he shall maintain no less than 0.2 mg/l free chlorine throughout the public water distribution system. When a particular sampling point has been shown to have a free chlorine residual less than 0.2 mg/l, the water at that location shall be retested as soon as practicable and in any event within one hour. If the original analysis is confirmed, this fact shall be reported to the State within 48 hours. Also, if the analysis is confirmed, a sample for coliform bacterial analysis must be collected from that sampling point as soon as practicable and preferably within one hour, and the results of such analysis reported to the State within 48 hours after the results are known to the supplier of water. Analyses for residual chlorine shall be made in accordance with "Standard Methods for the Examination of Water and Wastewater," 13th Ed., pp. 129–132. Compliance with the maximum contaminant levels for coliform bacteria shall be determined on the monthly mean or quarterly mean basis specified in § 141.14, including those samples taken as a result of failure to maintain the required chlorine residual level. The State may withdraw its approval of the use of chlorine residual substitution at any time.

§ 141.22 Turbidity sampling and analytical requirements.

(a) Samples shall be taken by suppliers of water for both community water systems and non-community water systems at a representative entry point(s) to the water distribution system at least once per day, for the purpose of making turbidity measurements to determine compliance with § 141.13. The measurement shall be made by the Nephelometric Method in accordance with the recommendations set forth in "Standard Methods for the Examination of Water and Wastewater," American Public Health Association, 13th Edition, pp. 350–353, or "Methods for Chemical Analysis of Water and Wastes," pp. 295–298, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(b) If the result of a turbidity analysis indicates that the maximum allowable limit has been exceeded, the sampling and measurement shall be confirmed by resampling as soon as practicable and preferably within one hour. If the repeat sample confirms that the maximum allowable limit has been exceeded, the supplier of water shall report to the State within 48 hours. The repeat sample shall be the sample used for the purpose of calculating the monthly average. If the monthly average of the daily samples exceeds the maximum allowable limit, or if the average of two samples taken on consecutive days exceeds 5 TU, the supplier of water shall report to the State and notify the public as directed in § 141.31 and § 141.32.

(c) Sampling for non-community water systems shall begin within two years after the effective date of this part.

(d) The requirements of this § 141.22 shall apply only to public water systems which use water obtained in whole or in part from surface sources.

§ 141.23 Inorganic chemical sampling and analytical requirements.

(a) Analyses for the purpose of determining compliance with § 141.11 are required as follows:

(1) Analyses for all community water systems utilizing surface water sources shall be completed within one year following the effective date of this part. These analyses shall be repeated at yearly intervals.

(2) Analyses for all community water systems utilizing only ground water sources shall be completed within two years following the effective date of this part. These analyses shall be repeated at three-year intervals.

(3) For non-community water systems, whether supplied by surface or ground water sources, analyses for nitrate shall be completed within two years following the effective date of this part. These analyses shall be repeated at intervals determined by the State.

(b) If the result of an analysis made pursuant to paragraph (a) indicates that the level of any contaminant listed in § 141.11 exceeds the maximum contaminant level, the supplier of water shall report to the State within 7 days and initiate three additional analyses at the same sampling point within one month.

(c) When the average of four analyses made pursuant to paragraph (b) of this section, rounded to the same number of significant figures as the maximum contaminant level for the substance in question, exceeds the maximum contaminant level, the supplier of water shall notify the State pursuant to § 141.31 and give notice to the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until the maximum contaminant level has not been exceeded in two successive samples or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(d) The provisions of paragraphs (b) and (c) of this section notwithstanding, compliance with the maximum contaminant level for nitrate shall be determined on the basis of the mean of two analyses. When a level exceeding the maximum contaminant level for nitrate is found, a second analysis shall be initiated within 24 hours, and if the mean of the two analyses exceeds the maximum contaminant level, the supplier of water shall report his findings to the State pursuant to § 141.31 and shall notify the public pursuant to § 141.32.

(e) For the initial analyses required by paragraph (a)(1), (2) or (3) of this section, data for surface waters acquired within one year prior to the effective date and data for ground waters acquired within 3 years prior to the effective date of this part may be substituted at the discretion of the State.

(f) Analyses conducted to determine compliance with § 141.11 shall be made in accordance with the following methods:

(1) Arsenic—Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 95–96, Environ-

mental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(2) Barium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes," pp. 97-98, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(3) Cadmium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes," pp. 101-103, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(4) Chromium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes," pp. 105-106, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(5) Lead—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes," pp. 112-113, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(6) Mercury—Flameless Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 118-126, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(7) Nitrate—Brucine Colorimetric Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 461-464, or Cadmium Reduction Method, "Methods for Chemical Analysis of Water and Wastes," pp. 201-206, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(8) Selenium—Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," p. 145, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(9) Silver—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater", 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes", p. 146, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(10) Fluoride—Electrode Method, "Standard Methods for the Examination of Water and Wastewater", 13th Edition, pp. 172-174, or "Methods for Chemical Analysis of Water and Wastes," pp. 65-67, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974, or Colorimetric Method with Preliminary Distillation, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 171-172 and 174-176, or "Methods for Chemical Analysis of Water and Wastes," pp. 59-60, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

§ 141.24 Organic chemical sampling and analytical requirements.

(a) An analysis of substances for the purpose of determining compliance with § 141.12 shall be made as follows:

(1) For all community water systems utilizing surface water sources, analyses shall be completed within one year following the effective date of this part. Samples analyzed shall be collected during the period of the year designated by the State as the period when contamination by pesticides is most likely to occur. These analyses shall be repeated at intervals specified by the State but in no event less frequently than at three year intervals.

(2) For community water systems utilizing only ground water sources, analyses shall be completed by those systems specified by the State.

(b) If the result of an analysis made pursuant to paragraph (a) of this section indicates that the level of any contaminant listed in § 141.12 exceeds the maximum contaminant level, the supplier of water shall report to the State within 7 days and initiate three additional analyses within one month.

(c) When the average of four analyses made pursuant to paragraph (b) of this section, rounded to the same number of significant figures as the maximum contaminant level for the substance in question, exceeds the maximum contaminant level, the supplier of water shall report to the State pursuant to § 141.31 and give notice to the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until the maximum contaminant level has not been exceeded in two successive samples or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(d) For the initial analysis required by paragraph (a) (1) and (2) of this section, data for surface water acquired within one year prior to the effective date of this part and data for ground water acquired within three years prior to the effective date of this part may be substituted at the discretion of the State.

(e) Analyses made to determine compliance with § 141.12(a) shall be made in accordance with "Method for Organochlorine Pesticides in Industrial Effluents," MDQARL, Environmental Protection Agency, Cincinnati, Ohio, November 28, 1973.

(f) Analyses made to determine compliance with § 141.12(b) shall be conducted in accordance with "Methods for Chlorinated Phenoxy Acid Herbicides in Industrial Effluents," MDQARL, Environmental Protection Agency, Cincinnati, Ohio, November 28, 1973.

§ 141.27 Alternative analytical techniques.

With the written permission of the State, concurred in by the Administrator of the U.S. Environmental Protection Agency, an alternative analytical technique may be employed. An alternative technique shall be acceptable only if it is substantially equivalent to the prescribed test in both precision and accuracy as it relates to the determination of compliance with any maximum contaminant level. The use of the alternative analytical technique shall not decrease the frequency of monitoring required by this part.

§ 141.28 Approved laboratories.

For the purpose of determining compliance with § 141.21 through § 141.27, samples may be considered only if they have been analyzed by a laboratory approved by the State except that measurements for turbidity and free chlorine residual may be performed by any person acceptable to the State.

§ 141.29 Monitoring of consecutive public water systems.

When a public water system supplies water to one or more other public water systems, the State may modify the monitoring requirements imposed by this part to the extent that the interconnection of the sysems jusifies treating them as a single system for monitoring purposes. Any modified monitoring shall be conducted pursuant to a schedule specified by the State and concurred in by the Administrator of the U.S. Environmental Protection Agency.

Subpart D—Reporting, Public Notification and Record Keeping

§ 141.31 Reporting requirements.

(a) Except where a shorter reporting period is specified in this part, the supplier of water shall report to the State within 40 days following a test, measurement or analysis required to be made by this part, the results of that test, measurement or analysis.

(b) The supplier of water shall report to the State within 48 hours the failure to comply with any primary drinking water regulation (including failure to comply with monitoring requirements) set forth in this part.

(c) The supplier of water is not required to report analytical results to the State in cases where a State laboratory performs the analysis and reports the results to the State office which would normally receive such notification from the supplier.

§ 141.32 Public notification.

(a) If a community water system fails to comply with an applicable maximum contaminant level established in Subpart B, fails to comply with an applicable testing procedure established in Subpart C of this part, is granted a variance or an exemption from an applicable maximum contaminant level, fails to comply with the requirements of any schedule prescribed pursuant to a variance or exemption, or fails to perform any monitoring required pursuant to Section 1445 (a) of the Act, the supplier of water shall notify persons served by the system of the failure or grant by inclusion of a notice in the first set of water bills of the system issued after the failure or grant and in any event by written notice within three months. Such notice shall be repeated at least once every three months so long as the system's failure continues or the variance or exemption remains in effect. If the system issues water bills less frequently than quarterly, or does not issue water bills, the notice shall be made by or supplemented by another form of direct mail.

(b) If a community water system has failed to comply with an applicable maximum contaminant level, the supplier of water shall notify the public of such failure, in addition to the notification required by paragraph (a) of this section, as follows:

(1) By publication on not less than three consecutive days in a newspaper or newspapers of general circulation in the area served by the system. Such notice shall be completed within fourteen days after the supplier of water learns of the failure.

(2) By furnishing a copy of the notice to the radio and television stations serving the area served by the system. Such notice shall be furnished within seven days after the supplier of water learns of the failure.

(c) If the area served by a community water system is not served by a daily newspaper of general circulation, notification by newspaper required by paragraph (b) of this section shall instead be given by publication on three consecutive weeks in a weekly newspaper of general circulation serving the area. If no weekly or daily newspaper of general circulation serves the area, notice shall be given by posting the notice in post offices within the area served by the system.

(d) If a non-community water system fails to comply with an applicable maximum contaminant level established in Subpart B of this part, fails to comply with an applicable testing procedure established in Subpart C of this part, is granted a variance or an exemption from an applicable maximum contaminant level, fails to comply with the requirement of any schedule prescribed pursuant to a variance or exemption or fails to perform any monitoring required pursuant to Section 1445(a) of the Act, the supplier of water shall given notice of such failure or grant to the persons served by the system. The form and manner of such notice shall be prescribed by the State, and shall insure that the public using the system is adequately informed of the failure or grant.

(e) Notices given pursuant to this section shall be written in a manner reasonably designed to inform fully the users of the system. The notice shall be conspicuous and shall not use unduly technical language, unduly small print or other methods which would frustrate the purpose of the notice. The notice shall disclose all material facts regarding the subject including the nature of the problem and, when appropriate, a clear statement that a primary drinking water regulation has been violated and any preventive measures that should be taken by the public. Where appropriate, or where designated by the State, bilingual notice shall be given. Notices may include a balanced explanation of the significance or seriousness to the public health of the subject of the notice, a fair explanation of steps taken by the system to correct any problem and the results of any additional sampling.

(f) Notice to the public required by this section may be given by the State on behalf of the supplier of water.

(g) In any instance in which notification by mail is required by paragraph (a) of this section but notification by newspaper or to radio or television stations is not required by paragraph (b) of this section, the State may order the supplier of water to provide notification by newspaper and to radio and television stations when circumstances make more immediate or broader notice appropriate to protect the public health.

§ 141.33 Record maintenance.

Any owner or operator of a public water system subject to the provisions of this part shall retain on its premises or at a convenient location near its premises the following records:

(a) Records of bacteriological analyses made pursuant to this part shall be kept for not less than 5 years. Records of chemical analyses made pursuant to this part shall be kept for not less than 10 years. Actual laboratory reports may be kept, or data may be transferred to tabular summaries, provided that the following information is included:

(1) The date, place, and time of sampling, and the name of the person who collected the sample;

(2) Identification of the sample as to whether it was a routine distribution system sample, check sample, raw or process water sample or other special purpose sample;

(3) Date of analysis;

(4) Laboratory and person responsible for performing analysis;

(5) The analytical technique/method used; and

(6) The results of the analysis.

(b) Records of action taken by the system to correct violations of primary drinking water regulations shall be kept for a period not less than 3 years after the last action taken with respect to the particular violation involved.

(c) Copies of any written reports, summaries or communications relating to sanitary surveys of the system conducted by the system itself, by a private consultant, or by any local, State or Federal agency, shall be kept for a period not less than 10 years after completion of the sanitary survey involved.

(d) Records concerning a variance or exemption granted to the system shall be kept for a period ending not less than 5 years following the expiration of such variance or exemption.

FROM p. 57332:

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 141

[FRL 1535-7]

Interim Primary Drinking Water Regulations; Amendments

AGENCY: Environmental Protection Agency (EPA).

→ **ACTION:** Final rule.

FROM p. 57332:

EFFECTIVE DATE: These amendments to the regulations will be effective August 27, 1980 except that sodium monitoring and reporting, determination of the types of materials in distribution systems, and monitoring and reporting corrosivity characteristics will be effective 18 months following the date of promulgation. The sodium and corrosion requirements must be completed within 12 months following the effective date.

FROM p. 57344:

9. Amending § 141.22(a) to read as follows and adding (e):

§ 141.22 Turbidity sampling and analytical requirements.

(a) Samples shall be taken by suppliers of water for both community and non-community water systems at a representative entry point(s) to the water distribution system at least once per day, for the purpose of making turbidity measurements to determine compliance with § 141.13. If the State determines that a reduced sampling frequency in a non-community system will not pose a risk to public health, it can reduce the required sampling frequency. The option of reducing the turbidity frequency shall be permitted only in those public water systems that practice disinfection and which maintain an active residual disinfectant in the distribution system, and in those cases where the State has indicated in writing that no unreasonable risk to health existed under the circumstances of this option. The turbidity measurements shall be made by the Nephelometric Method in accordance with the recommendations set forth in "Standard Methods for Examination of Water and Wastewater," American Public Health Association, 14th Edition, pp. 132-134; or Method 180.1,1-Nephrometric Method.

* * * * *

(e) The State has the authority to determine compliance or initiate enforcement action based upon analytical results or other information compiled by their sanctioned representatives and agencies.

10. Amending § 141.23(a)(3), adding (a)(4) and amending (f) (1) through (10) to read as follows:

§ 141.23 Inorganic chemical sampling and analytical requirements.

(a) * * *

(3) For non-community water systems, whether supplied by surface or ground sources, analyses for nitrate shall be completed by December 24, 1980. These analyses shall be repeated at intervals determined by the State.

(4) The State has the authority to determine compliance or initiate enforcement action based upon analytical results and other information compiled by their sanctioned representatives and agencies.

* * * * *

(f) * * *

(1) Arsenic—Method [1] 206.2, Atomic Absorption Furnace Technique; or Method [1] 206.3, or Method [4] D2972-78A, or Method [3] 301.A VII, pp. 159-162, or Method [3] I-1062-78, pp. 61-63, Atomic Absorption—Gaseous Hydride; or Method [1] 206.4, or Method [4] D-2972-78A, or Method [2] 404-A and 404-B(4), Spectrophotometric, Silver Diethyldithiocarbamate.

(2) Barium—Method [1] 208.1, or Method [2] 301-A IV, pp. 152-155, Atomic Absorption—Direct Aspiration; or Method [1] 208.2, Atomic Absorption Furnace Technique.

(3) Cadmium—Method [1] 213.1, or Method [4] 3557-78A or B, or Method [2] 301-A II or III, pp. 148-152, Atomic Absorption—Direct Aspiration; or Method [1] 213.2, Atomic Absorption Furnace Technique.

(4) Chromium—Method [1] 218.1, or Method [4] D-1687-77D, or Method [2] 301-A II or III, pp. 148-152, Atomic Absorption—Direct Aspiration; or Chromium—Method [1] 218.2, Atomic Absorption Furnace Technique.

(5) Lead—Method [1] 239.1, or Method [4] D-3559-78A or B, or Method [2] 301-A II or III, pp. 148-152, Atomic Absorption—Direct Aspiration; or Method [1] 239.2, Atomic Absorption Furnace Technique.

(6) Mercury—Method [1] 245.1, or Method [4] D-3223-79, or Method [2] 301-A VI, pp. 156-159, Manual Cold Vapor Technique; or Method [1] 245.2, Automated Cold Vapor Technique.

(7) Nitrate—Method [1] 352.1, or Method [4] D-992-71, or Method [2] 419-D, pp. 427-429, Colorimetric Brucine; or Method [1] 353.3, or Method [4] D-3867-79B, or Method [2] 419-C, pp. 423-427, Spectrometric, Cadmium Reduction; Method [1] 353.1, Automated Hydrazine Reduction; or Method [1] 353.2, or Method [4] D-3867-79A, or Method [2] 605, pp. 620-624, Automated Cadmium Reduction.

(8) Selenium—Method [1] 270.2, Atomic Absorption Technique; or Method [1] 270.3; or Method [3] I-1667-78, pp. 237-239, or Method [4] D-3859-79, or Method [2] 301-A VII, pp. 159-162, Hydride Generation—Atomic Absorption Spectrophotometry.

(9) Silver—Method [1] 272.1, or Method [2] 301-A II, Atomic Absorption—Direct Aspiration; or Method [1] 272.2, Atomic Absorption Techniques Furnace Technique.

(10) Fluoride—Electrode Method, or SPADNS Method, Method [2] 414-B and C, pp. 391-394, or Method [1] 340.1, "Colorimetric SPADNS with Bellack Distillation," or Method [1] 340.2, "Potentimetric Ion Selective Electrode;" or ASTM Method [4] D1179-72; or Colorimetric Method with Preliminary Distillation, Method [2] 603, Automated Complexone Method (Alizarin Fluoride Blue) pp. 614-616; or Automated Electrode Method, "Fluoride in Water and Wastewater," Industrial Method #380-75WE, Technicon Industrial Systems, Tarrytown, New York 10591, February 1976, or "Fluoride in Water

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[1] "Methods of Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268 (EPA-600/4-79-020), March 1979. Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268. For approved analytical procedures for metals, the technique applicable to total metals must be used.

[2] "Standard Methods for the Examination of Water and Wastewater," 14th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1976.

[3] Techniques of Water—Resources Investigation of the United States Geological Survey, Chapter A-1, "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," Book 5, 1979, Stock #024-001-03177-9. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[4] Annual Book of ASTM Standards, part 31 Water, American Society for Testing and Materials, 1976, Race Street, Philadelphia, Pennsylvania 19103.

and Wastewater Industrial Method #129–71W," Technicon Industrial Systems, Tarrytown, New York 10591, December 1972; or Fluoride, Total, Colorimetic, Zirconium—Eriochrome Cyanine R Method [3]—I–3325–78, pp. 365–367.

11. Amending §141.24(a)(3), (e) and (f) to read as follows:

§ 141.24 Organic chemical sampling and analytical requirements.

(a) * * *

(3) The State has the authority to determine compliance or initiate enforcement action based upon analytical results and other information compiled by their sanctioned representatives and agencies.

* * * * *

(e) Analysis made to determine compliance with § 141.12(a) shall be made in accordance with "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water," available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268; or "Organochlorine Pesticides in Water," 1977 Annual Book of ASTM Standards, part 31, Water, Method D3088; or Metnod 509–A, pp. 555–565; [2] or Gas Chromatographic Methods for Analysis of Organic Substances in Water,[3] USGS, Book 5, Chapter A–5, pp. 24–39.

(f) Analysis made to determine compliance with § 141.12(b) shall be conducted in accordance with "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water," available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268; or "Chlorinated Phenoxy Acid Herbicides in Water," 1977 Annual Book of ASTM Standards, part 31, Method D3478; or Method 509–B, pp. 555–5692; [2] or Gas Chromatographic Methods for Analysis of Organic Substances in Water,[3] USGS, Book 5, Chapter A–3, pp. 24–39.

§ 141.25 [Amended]

12. Amending § 141.25 to add (e):

(e) The State has the authority to determine compliance or initiate enforcement action based upon analytical results or other information compiled by their sanctioned representatives and agencies.

13. Amending § 141.27(a) to read as follows:

§ 141.27 Alternate analytical techniques.

(a) With the written permission of the State, concurred in by the Administrator of the U.S. EPA, an alternate analytical technique may be employed. An alternate technique shall be accepted only if it is substantially equivalent to the prescribed test in both precision and accuracy as it relates to the determination of compliance with any MCL. The use of the alternate analytical technique shall not decrease the frequency of monitoring required by this part.

14. Amending § 141.28 to read as follows:

§ 141.28 Approved laboratories.

(a) For the purpose of determining compliance with § 141.21 through § 141.27, samples may be considered only if they have been analyzed by a laboratory approved by the State except that measurements for turbidity, free chlorine residual, temperature and pH may be performed by any person acceptable to the State.

(b) Nothing in this Part shall be construed to preclude the State or any duly designated representative of the State from taking samples or from using the results from such samples to determine compliance by a supplier of water with the applicable requirements of this Part.

15. Amending § 141.31 (a) and (c) and adding paragraphs (d) and (e) to read as follows:

§ 141.31 Reporting requirements.

(a) Except where a shorter period is specified in this part, the supplier of water shall report to the State the results of any test measurement or analysis required by this part within (A) the first ten days following the month in which the result is received or (B) the first ten days following the end of the required monitoring period as stipulated by the State, whichever of these is shortest.

* * * * *

(d) The water supply system, within ten days of completion of each public notification required pursuant to § 141.32, shall submit to the State a representative copy of each type of notice distributed, published, posted, and/or made available to the persons served by the system and/or to the media.

(e) The water supply system shall submit to the State within the time stated in the request copies of any records required to be maintained under § 141.33 hereof or copies of any documents then in existence which the State or the Administrator is entitled to inspect pursuant to the authority of § 1445 of the Safe Drinking Water Act or the equivalent provisions of State law.

16. Amending § 141.32 (b)(3) and (d) to read as follows:

§ 141.32 Public notification.

* * * * *

(b) * * *

(3) Except that the requirements of this subsection (b) may be waived by the State if it determines that the violation has been corrected promptly after discovery, the cause of the violation has been eliminated, and there is no longer a risk to public health.

* * * * *

(d) If a non-community water system fails to comply with an applicable MCL established in Subpart B of this part, fails to comply with an applicable testing procedure established in Subpart C of this part, is granted a variance or an exemption from an applicable MCL, fails to comply with the requirements of any schedule prescribed pursuant to a variance or exemption, or fails to perform any monitoring requirement pursuant to section 1445(a) of the Act, the supplier of water shall give notices by continuous posting of such failure or granting of a variance or exemption to the persons served by the system as long as the failure or granting of a variance or exemption continues. The form and manner for such notices shall be prescribed by the State and shall ensure that the public using the system is adequately informed of the failure or granting of the variance or exemption.

17. Amending Subpart E to read as follows:

Subpart E—Special Monitoring Regulations for Organic Chemicals and Otherwise Unregulated Contaminants

§ 141.41 Special monitoring for sodium.

(a) Suppliers of water for community public water systems shall collect and analyze one sample per plant at the entry point of the distribution system for the determination of sodium concentration levels; samples must be collected and analyzed annually for systems utilizing surface water sources in whole or in part, and at least every three years for systems utilizing solely ground water sources. The minimum number of samples required to be taken by the system shall be based on the number of treatment plants used by the system, except that multiple wells drawing raw water from a single aquifer may, with the State approval, be considered one treatment plant for determining the minimum number of samples. The supplier of water may be required by the State to collect and analyze water samples for sodium more frequently in locations where the sodium content is variable.

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[3] Techniques of Water—Resources Investigation of the United States Geological Survey, Chapter A–3, "Methods for Analysis of Organic Substances in Water," Book 5, 1972, Stock #2401–1227. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

(b) The supplier of water shall report to EPA and/or the State the results of the analyses for sodium within the first 10 days of the month following the month in which the sample results were received or within the first 10 days following the end of the required monitoring period as stipulated by the State, whichever of these is first. If more than annual sampling is required the supplier shall report the average sodium concentration within 10 days of the month following the month in which the analytical results of the last sample used for the annual average was received. The supplier of water shall not be required to report the results to EPA where the State has adopted this regulation and results are reported to the State. The supplier shall report the results to EPA where the State has not adopted this regulation.

(c) The supplier of water shall notify appropriate local and State public health officials of the sodium levels by written notice by direct mail within three months. A copy of each notice required to be provided by this paragraph shall be sent to EPA and/or the State within 10 days of its issuance. The supplier of water is not required to notify appropriate local and State public health officials of the sodium levels where the State provides such notices in lieu of the supplier.

(d) Analyses for sodium shall be performed by the flame photometric method in accordance with the procedures described in "Standard Methods for the Examination of Water and Wastewater," 14th Edition, pp. 250–253; or by Method 273.1, Atomic Absorption—Direct Aspiration or Method 273.2, Atomic Absorption—Graphite Furnace, in "Methods for Chemical Analysis of Water and Waste," EMSL, Cincinnati, EPA, 1979; or by Method D1428–64(a) in Annual Book of ASTM Standards, part 31, Water.

18. Adding a § 141.42 to read as follows:

§ 141.42 Special monitoring for corrosivity characteristics.

(a) Suppliers of water for community public water systems shall collect samples from a representative entry point to the water distribution system for the purpose of analysis to determine the corrosivity characteristics of the water.

(1) The supplier shall collect two samples per plant for analysis for each plant using surface water sources wholly or in part or more if required by the State; one during mid-winter and one during mid-summer. The supplier of the water shall collect one sample per plant for analysis for each plant using ground water sources or more if required by the State. The minimum number of samples required to be taken by the system shall be based on the number of treatment plants used by the system, except that multiple wells drawing raw water from a single aquifer may, with the State approval, be considered one treatment plant for determining the minimum number of samples.

(2) Determination of the corrosivity characteristics of the water shall include measurement of pH, calcium hardness, alkalinity, temperature, total dissolved solids (total filterable residue), and calculation of the Langelier Index in accordance with paragraph (c) below. The determination of corrosivity characteristics shall only include one round of sampling (two samples per plant for surface water and one sample per plant for ground water sources). However, States may require more frequent monitoring as appropriate. In addition, States have the discretion to require monitoring for additional parameters which may indicate corrosivity characteristics, such as sulfates and chlorides. In certain cases, the Aggressive Index, as described in paragraph (c), can be used instead of the Langelier Index; the supplier shall request in writing to the State and the State will make this determination.

(b) The supplier of water shall report to EPA and/or the State the results of the analyses for the corrosivity characteristics within the first 10 days of the month following the month in which the sample results were received. If more frequent sampling is required by the State, the supplier can accumulate the data and shall report each value within 10 days of the month following the month in which the analytical results of the last sample was received. The supplier of water shall not be required to report the results to EPA where the State has adopted this regulation and results are reported to the State.

(c) Analyses conducted to determine the corrosivity of the water shall be made in accordance with the following methods:

(1) Langelier Index—"Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 203, pp. 61–63.

(2) Aggressive Index—"AWWA Standard for Asbestos-Cement Pipe, 4 in. through 24 in. for Water and Other Liquids," AWWA C400–77, Revision of C400–75, AWWA, Denver, Colorado.

(3) Total Filtrable Residue—"Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 208B, pp. 92–93; or "Methods for Chemical Analysis of Water and Wastes," Method 160.1.

(4) Temperature—"Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 212, pp. 125–126.

(5) Calcium hardness—EDTA Titrimetric Method "Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 309B, pp. 202–206; or "Annual Book of ASTM Standards," Method D1126–67 (8).

(6) Alkalinity—Methyl Orange and paint pH 4.5. "Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 403, pp. 278–281; or "Annual Book of ASTM Standards," Method D1067–70B; or "Methods for Chemical Analysis of Water and Wastes," Method 310.1.

(7) pH—"Standard Methods for the Examination of Water and Wastewater," 14th Edition, Method 424, pp. 460–465; or "Methods for Chemical Analysis of Water and Wastes," Method 150.1; or "Annual Book of ASTM Standards," Method D129378 A or B.

(8) Chloride—Potentiometric Method, "Standard Methods for the Examination of Water and Wastewater," 14th Edition, p. 306.

(9) Sulfate—Turbidimetric Method, "Methods for Chemical Analysis of Water and Wastes," pp. 277–278, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 334–335, 14th Edition, pp. 496–498.

(d) Community water supply systems shall identify whether the following construction materials are present in their distribution system and report to the State:

- Lead from piping, solder, caulking, interior lining of distribution mains, alloys and home plumbing.
- Copper from piping and alloys, service lines, and home plumbing.
- Galvanized piping, service lines, and home plumbing.
- Ferrous piping materials such as cast iron and steel.
- Asbestos cement pipe.

In addition, States may require identification and reporting of other materials of construction present in distribution systems that may contribute contaminants to the drinking water, such as:

- Vinyl lined asbestos cement pipe.
- Coal tar lined pipes and tanks.

Appendix A—Response to Public Comments

Comments submitted to the Agency and statements presented at the public hearing in

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 141

[FRL 1312-2]

National Interim Primary Drinking Water Regulations; Control of Trihalomethanes in Drinking Water

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final Rule.

SUMMARY: This amendment to the National Interim Primary Drinking Water Regulations establishes a Maximum Contaminant Level (MCL) of 0.10 mg/l and associated monitoring and reporting requirements for total trihalomethanes (TTHMs), including chloroform, that are introduced into drinking water by the reaction of naturally occurring substances with chlorine in the course of water treatment. The proposed requirement to utilize granular activated carbon (GAC) or equivalent technology in those public water systems subject to significant contamination by synthetic organic chemicals has been separated from this promulgation and will be reproposed for additional public comment in the near future.

EFFECTIVE DATES: For community water systems serving 75.000 or more persons. monitoring must begin 1 year following promulgation and the effective date of the MCL is 2 years following promulgation. For community water systems serving 10.000 to 75.000 persons. monitoring must begin within 3 years from the date of promulgation and the effective date of the MCL is 4 years from the date of promulgation. Effective immediately. systems that plan to make significant modifications to their treatment processes for the purpose of complying with the TTHM MCL are required to seek and obtain State approval of their treatment modification plans.

FOR FURTHER INFORMATION CONTACT: Joseph A. Cotruvo. Director. Criteria and Standards Division. Office of Drinking Water (WH-550). Environmental Protection Agency. 401 M Street. S.W.. Washington. D.C. 20460. (202-472-5016).

SUPPLEMENTARY INFORMATION:

History of Rulemaking

On July 14. 1976. EPA published an Advance Notice of Proposed Rulemaking (ANPRM). entitled "Control Options for Organic Chemicals in Drinking Water" (41 FR 28991 *et seq.*). The ANPRM summarized the many facets of the issue of organic chemicals in drinking water including the legislative background. health effects data. the state of available control technology and costs. Advantages and disadvantages of various regulatory and non-regulatory options were examined. and the ANPRM solicited comments and information regarding the problem and options presented. On February 9. 1978. the EPA published a proposed rule (43 FR 5756. *et seq.*) To amend the National Interim Primary Drinking Water Regulations to include an MCL and associated monitoring and reporting requirements for TTHMs. At the same time. a requirement for the use of GAC or equivalent technology was proposed for application to those drinking water sources subject to significant contamination by synthetic organic chemicals of industrial origin. Subsequently. on July 6. 1978. EPA published a Supplemental Notice of Proposed Rulemaking (43 FR 29135. *et seq.*) soliciting comment on EPA's reassessment of the economic impact analysis for the proposal. providing additional documentation in support of the proposal. clarifying certain aspects concerning the effects of organic chemicals in drinking water. and extending the public comment period from July 31. 1978. to September 1. 1978.

The two Federal Register Notice preambles and the supporting documentation cited therein provided a detailed discussion of EPA's rationale for proposing controls on organic chemicals in drinking water. The subjects covered included: assessments of the sources and occurrences of. and human exposure to. THMs and other organic chemicals in drinking water: discussion of the toxicology and epidemiology studies that relate to possible human health risks: rationale for the selection of the MCL for TTHMs and associated requirements: and a discussion of the control technology. economic impact and air pollution and energy impacts of the proposal. EPA's analyses of these subjects have been revised to incorporate information gained during the public comment period.

A total of 598 written comments were received in response to the proposed regulations of which 391 addressed the subject of THMs. In a number of cases the commenters confused the two different regulations being proposed for organic chemical control. For example. some commenters incorrectly assumed that GAC was proposed as the requirement for control of THMs and objected accordingly.

Public hearings were held between March and July. 1978. in Miami. Florida: New Orleans. Louisiana: Boston. Massachusetts: Los Angeles. California: St. Louis. Missouri: Louisville. Kentucky: Washington. D.C. and Dallas. Texas. A total of 259 witnesses testified at the public hearings. and of these. 157 commented on the proposed regulations for THMs. Commenters included water utilities. state and local officials. public interest groups. federal health regulatory and research agencies. engineering consulting firms and individual citizens and scientists. In addition. there were 496 communications from members of Congress. and both the House and Senate Appropriations Committees. and the Council on Wage and Price Stability offered comments on the proposed regulations. The National Drinking Water Advisory Council was also consulted for their comments on the regulations. A number of the comments were duplicative. in that often the same persons or organizations submitted both written and oral comments and such comments often induced inquiries from members of Congress on the same subject. EPA has thoroughly considered all comments received in formulating the final regulations. A detailed breakdown of the comments and the Agency's responses to them are attached as Appendices.

Legal Authority

These final regulations are issued under the authority of the Safe Drinking Water Act. as amended (SDWA). 42 U.S.C. 300f *et seq.*. specifically. sections 1401. 1412. 1445 and 1450. They constitute amendments to the National Interim Primary Drinking Water Regulations (NIPDWR). 40 CFR Part 141. as authorized by Section 1412(a)(1).

As noted in the preamble to the proposed regulations (43 FR at 5759). EPA considered establishing these regulations as Revised Primary Drinking Water Regulations but concluded that they would be more appropriate as amendments to the NIPDWR. This means that the feasibility of control measures under the NIPDWR must be adjudged to have been available as of December. 1974. when the SDWA was enacted. As prescribed by Section 1412(a)(2). these Interim Regulations protect health to the extent feasible. using technology. treatment techniques. and other means which the Administrator determines are generally available (taking costs into consideration) on the date of enactment (of the SDWA).

Although Congress clearly contemplated the comprehensive control of organic chemical contaminants in the

Page 8-44 intentionally
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regulation not subject to the procedural requirements of Executive Order 12044.

Dated: November 5, 1979.

Douglas M. Costle,
Administrator.

Accordingly, Part 141, Title 40 of the Code of Federal Regulations is hereby amended as follows:

1. By amending § 141.2 to include the following new paragraphs (p) through (t):

§ 141.2 Definitions.

* * * * *

(p) "Halogen" means one of the chemical elements chlorine, bromine or iodine.

(q) "Trihalomethane" (THM) means one of the family of organic compounds, named as derivatives of methane, wherein three of the four hydrogen atoms in methane are each substituted by a halogen atom in the molecular structure.

(r) "Total trihalomethanes" (TTHM) means the sum of the concentration in milligrams per liter of the trihalomethane compounds (trichloromethane [chloroform], dibromochloromethane, bromodichloromethane and tribromomethane [bromoform]), rounded to two significant figures.

(s) "Maximum Total Trihalomethane Potential (MTP)" means the maximum concentration of total trihalomethanes produced in a given water containing a disinfectant residual after 7 days at a temperature of 25° C or above.

(t) "Disinfectant" means any oxidant, including but not limited to chlorine, chlorine dioxide, chloramines, and ozone added to water in any part of the treatment or distribution process, that is intended to kill or inactivate pathogenic microorganisms.

2. By revising § 141.6 to read as follows:

§ 141.6 Effective dates.

(a) Except as provided in paragraph (b) of this section, the regulations set forth in this part shall take effect on June 24, 1977.

(b) The regulations for total trihalomethanes set forth in § 141.12(c) shall take effect 2 years after the date of promulgation of these regulations for community water systems serving 75,000 or more individuals, and 4 years after the date of promulgation for communities serving 10,000 to 74,999 individuals.

3. By revising the introductory paragraph and adding a new paragraph (c) in § 141.12 to read as follows:

§ 141.12 Maximum contaminant levels for organic chemicals.

The following are the maximum contaminant levels for organic chemicals. The maximum contaminant levels for organic chemicals in paragraphs (a) and (b) of this section apply to all community water systems. Compliance with the maximum contaminant levels in paragraphs (a) and (b) is calculated pursuant to § 141.24. The maximum comtaminant level for total trihalomethanes in paragraph (c) of this section applies only to community water systems which serve a population of 10,000 or more individuals and which add a disinfectant (oxidant) to the water in any part of the drinking water treatment process. Compliance with the maximum contaminant level for total trihalomethanes is calculated pursuant to § 141.30.

* * * * *

(c) Total trihalomethanes (the sum of the concentrations of bromodichloromethane, dibromochloromethane, tribromomethane (bromoform) and trichloromethane (chloroform)) 0.10 mg/l.

4. By revising the title, the introductory text of paragraph (a) and paragraph (b) of § 141.24 to read as follows:

§ 141.24 Organic chemicals other than total trihalomethanes, sampling and analytical requirements.

(a) An analysis of substances for the purpose of determining compliance with § 141.12(a) and § 141.12(b) shall be made as follows:

(b) If the result of an analysis made pursuant to paragraph (a) of this section indicates that the level of any contaminant listed in § 141.24 (a) and (b) exceeds the maximum contaminant level, the supplier of water shall report to the State within 7 days and initiate three additional analyses within one month.

5. By adding a new § 141.30 to read as follows:

§ 141.30 Total trihalomethanes sampling, analytical and other requirements.

(a) Community water system which serve a population of 10,000 or more individuals and which add a disinfectant (oxidant) to the water in any part of the drinking water treatment process shall analyze for total trihalomethanes in accordance with this section. For systems serving 75,000 or more individuals, sampling and analyses shall begin not later than 1 year after the date of promulgation of this regulation. For systems serving 10,000 to 74,999 individuals, sampling and analyses shall begin not later than 3 years after the date of promulgation of this regulation. For the purpose of this section, the minimum number of samples required to be taken by the system shall be based on the number of treatment plants used by the system, except that multiple wells drawing raw water from a single aquifer may, with the State approval, be considered one treatment plant for determining the minimum number of samples. All samples taken within an established frequency shall be collected within a 24-hour period.

(b)(1) For all community water systems utilizing surface water sources in whole or in part, and for all community water systems utilizing only ground water sources that have not been determined by the State to qualify for the monitoring requirements of paragraph (c) of this section, analyses for total trihalomethanes shall be performed at quarterly intervals on at least four water samples for each treatment plant used by the system. At least 25 percent of the samples shall be taken at locations within the distribution system reflecting the maximum residence time of the water in the system. The remaining 75 percent shall be taken at representative locations in the distribution system, taking into account number of persons served, different sources of water and different treatment methods employed. The results of all analyses per quarter shall be arithmetically averaged and reported to the State within 30 days of the system's receipt of such results. Results shall also be reported to EPA until such monitoring requirements have been adopted by the State. All samples collected shall be used in the computation of the average, unless the analytical results are invalidated for technical reasons. Sampling and analyses shall be conducted in accordance with the methods listed in paragraph (e) of this section.

(2) Upon the written request of a community water system, the monitoring frequency required by paragraph (b)(1) of this section may be reduced by the State to a minimum of one sample analyzed for TTHMs per quarter taken at a point in the distribution system reflecting the maximum residence time of the water in the system, upon a written determination by the State that the data from at least 1 year of monitoring in accordance with paragraph (b)(1) of this section and local conditions demonstrate that total trihalomethane concentrations will be consistently below the maximum contaminant level.

(3) If at any time during which the reduced monitoring frequency prescribed under this paragraph applies, the results from any analysis exceed 0.10 mg/l of TTHMs and such results are confirmed by at least one check sample taken promptly after such results are received, or if the system makes any significant change to its source of water or treatment program, the system shall immediately begin monitoring in accordance with the requirements of paragraph (b)(1) of this section, which monitoring shall continue for at least 1 year before the frequency may be reduced again. At the option of the State, a system's monitoring frequency may and should be increased above the minimum in those cases where it is necessary to detect variations of TTHM levels within the distribution system.

(c)(1) Upon written request to the State, a community water system utilizing only ground water sources may seek to have the monitoring frequency required by subparagraph (1) of paragraph (b) of this section reduced to a minimum of one sample for maximum TTHM potential per year for each treatment plant used by the system taken at a point in the distribution system reflecting maximum residence time of the water in the system. The system shall submit to the State the results of at least one sample analyzed for maximum TTHM potential for each treatment plant used by the system taken at a point in the distribution system reflecting the maximum residence time of the water in the system. The system's monitoring frequency may only be reduced upon a written determination by the State that, based upon the data submitted by the system, the system has a maximum TTHM potential of less than 0.10 mg/l and that, based upon an assessment of the local conditions of the system, the system is not likely to approach or exceed the maximum contaminant level for total TTHMs. The results of all analyses shall be reported to the State within 30 days of the system's receipt of such results. Results shall also be reported to EPA until such monitoring requirements have been adopted by the State. All samples collected shall be used for determining whether the system must comply with the monitoring requirements of paragraph (b) of this section, unless the analytical results are invalidated for technical reasons. Sampling and analyses shall be conducted in accordance with the methods listed in paragraph (e) of this section.

(2) If at any time during which the reduced monitoring frequency prescribed under paragraph (c)(1) of this section applies, the results from any analysis taken by the system for maximum TTHM potential are equal to or greater than 0.10 mg/l, and such results are confirmed by at least one check sample taken promptly after such results are received, the system shall immediately begin monitoring in accordance with the requirements of paragraph (b) of this section and such monitoring shall continue for at least one year before the frequency may be reduced again. In the event of any significant change to the system's raw water or treatment program, the system shall immediately analyze an additional sample for maximum TTHM potential taken at a point in the distribution system reflecting maximum residence time of the water in the system for the purpose of determining whether the system must comply with the monitoring requirements of paragraph (b) of this section. At the option of the State, monitoring frequencies may and should be increased above the minimum in those cases where this is necessary to detect variation of TTHM levels within the distribution system.

(d) Compliance with § 141.12(c) shall be determined based on a running annual average of quarterly samples collected by the system as prescribed in subparagraphs (1) or (2) of paragraph (b) of this section. If the average of samples covering any 12 month period exceeds the Maximum Contaminant Level, the supplier of water shall report to the State pursuant to § 141.31 and notify the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(e) Sampling and analyses made pursuant to this section shall be conducted by one of the following EPA approved methods:

(1) "The Analysis of Trihalomethanes in Finished Waters by the Purge and Trap Method," Method 501.1, EMSL, EPA Cincinnati, Ohio.

(2) "The Analysis of Trihalomethanes in Drinking Water by Liquid/Liquid Extraction,"Method 501.2, EMSL, EPA Cincinnati, Ohio.

Samples for TTHM shall be dechlorinated upon collection to prevent further production of Trihalomethanes, according to the procedures described in the above two methods. Samples for maximum TTHM potential should not be dechlorinated, and should be held for seven days at 25° C prior to analysis.

according to the procedures described in the above two methods.

(f) Before a community water system makes any significant modifications to its existing treatment process for the purposes of achieving compliance with § 141.12(c), such system must submit and obtain State approval of a detailed plan setting forth its proposed modification and those safeguards that it will implement to ensure that the bacteriological quality of the drinking water served by such system will not be adversely affected by such modification. Each system shall comply with the provisions set forth in the State-approved plan. At a minimum, A State approved plan shall require the system modifying its disinfection practice to:

(1) Evaluate the water system for sanitary defects and evaluate the source water for biological quality;

(2) Evaluate its existing treatment practices and consider improvements that will minimize disinfectant demand and optimize finished water quality throughout the distribution system;

(3) Provide baseline water quality survey data of the distribution system. Such data should include the results from monitoring for coliform and fecal coliform bacteria, fecal streptococci, standard plate counts at 35° C and 20° C, phosphate, ammonia nitrogen and total organic carbon. Virus studies should be required where source waters are heavily contaminated with sewage effluent;

(4) Conduct additional monitoring to assure continued maintenance of optimal biological quality in finished water, for example, when chloramines are introduced as disinfectants or when pre-chlorination is being discontinued. Additional monitoring should also be required by the State for chlorate, chlorite and chlorine dioxide when chlorine dioxide is used as a disinfectant. Standard plate count analyses should also be required by the State as appropriate before and after any modifications;

(5) Demonstrate an active disinfectant residual throughout the distribution system at all times during and after the modification.

This paragraph (f) shall become effective on the date of its promulgation.

Appendix A—Summary of Public Comments and EPA Responses on Proposed Amendments to the National Interim Primary Drinking Water Regulations for Control of Trihalomethanes in Drinking Water

The following is a summary and discussion of the principal public comments to EPA's proposed regulations for the control of ●●●

follows. These regulations will take effect January 19, 1981.

Dated: July 12, 1979.

Douglas M. Costle,
Administrator.

PART 143—NATIONAL SECONDARY DRINKING WATER REGULATIONS

Sec.
143.1 Purpose.
143.2 Definitions.
143.3 Secondary maximum contaminant levels.
143.4 Monitoring.

Authority: Section 1412(c) of the Safe Drinking Water Act, as amended (42 U.S.C. 300g–1(c))

§ 143.1 Purpose.

This part establishes National Secondary Drinking Water Regulations pursuant to Section 1412 of the Safe Drinking Water Act, as amended (42 U.S.C. 300g–1). These regulations control contaminants in drinking water that primarily affect the aesthetic qualities relating to the public acceptance of drinking water. At considerably higher concentrations of these contaminants, health implications may also exist as well as aesthetic degradation. The regulations are not Federally enforceable but are intended as guidelines for the States.

§ 143.2 Definitions.

(a) "Act" means the Safe Drinking Water Act as amended (42 U.S.C. 300f *et seq.*).

(b) "Contaminant" means any physical, chemical, biological, or radiological substance or matter in water.

(c) "Public water system" means a system for the provision to the public of piped water for human consumption, if such a system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system. A public water system is either a "community water system" or a "non-community water system."

(d) "State" means the agency of the State government which has jurisdiction over public water systems.

(e) "Supplier of water" means any person who owns or operates a public water system.

(f) "Secondary Maximum Contaminant Levels" means SMCLs which apply to public water systems and which, in the judgement of the Administrator, are requisite to protect the public welfare. The SMCL means the maximum permissible level of a contaminant in water which is delivered to the free flowing outlet of the ultimate user of public water system. Contamimants added to the water under circumstances controlled by the user, except those resulting from corrosion of piping and plumbing caused by water quality, are excluded from this definition.

§ 143.3 Secondary Maximum Contaminant Levels.

The Secondary Maximum Contaminant Levels for public water systems are as follows:

Contaminant:	Level
Chloride	250 mg/l.
Color	15 color units
Copper	1 mg/l.
Corrosivity	Noncorrosive
Foaming agents	0.5 mg/l.
Iron	0.3 mg/l.
Manganese	0.05 mg/l.
Odor	3 threshold odor number.
pH	6.5–8.5.
Sulfate	250 mg/l.
Total dissolved solids (TDS)	500 mg/l.
Zinc	5 mg/l.

These levels represent reasonable goals for drinking water quality. The States may establish higher or lower levels which may be appropriate dependent upon local conditions such as unavailability of alternate source waters or other compelling factors, provided that public health and welfare are not adversely affected.

§ 143.4 Monitoring.

(a) It is recommended that the parameters in these regulations should be monitored at intervals no less frequent than the monitoring performed for inorganic chemical contaminants listed in the National Interim Primary Drinking Water Regulations as applicable to community water systems. More frequent monitoring would be appropriate for specific parameters such as pH, color, odor or others under certain circumstances as directed by the State.

(b) Analyses conducted to determine compliance with § 143.3 should be made in accordance with the following methods:

(1) Chloride—Potentiometric Method, "Standard Methods for the Examination of Water and Wastewater," 14th Edition, p. 306.

(2) Color—Platinum-Cobalt Method, "Methods for Chemical Analysis of Water and Wastes," p. 36–38, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 160–162, 14th Edition, p. 64–66.

(3) Copper—Atomic Adsorption Method, "Methods for Chemical Analysis of Water and Wastes", pp. 108–109, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, 14th Edition, p. 144–147.

(4) Foaming Agents—Methylene Blue Method, "Methods for Chemical Analysis of Water and Wastes," pp. 157–158, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 339–342, 14th Edition, p. 600.

(5) Iron—Atomic Adsorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 110–111, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, 14th Edition, p. 144–147.

(6) Manganese—Atomic Adsorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 116–117, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, 14th Edition, p. 144–147.

(7) Odor—Consistent Series Method, "Methods for Chemical Analysis of Water and Wastes," pp. 287–294, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 248–254, 14th Edition, p. 75–82.

(8) pH—Glass Electrode Method, "Methods for Chemical Analysis of Water and Wastes," pp. 239–240, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 276–281, 14th Edition, pp. 460–465.

(9) Sulfate—Turbidimetric Method, "Methods for Chemical Analysis of Water and Wastes," pp. 277–278, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th

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Edition, pp. 334–335, 14th Edition, p. 496–498.

(10) Total Dissolved Solids—Total Residue Methods, "Methods for Chemical Analysis of Water and Wastes," pp. 270–271, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 288–290, 14th Edition, p. 91–92.

(11) Zinc—Atomic Adsorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 155–156, EPA, Office of Technology Transfer, Washington, D.C. 20460, 1974, or "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210–215, 14th Edition, p. 144–147.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Laboratory Safety Practices

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 30 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant must use correct laboratory safety practices during the laboratory sessions in this course and in his/her daily work situation.

V. ENTRY LEVEL BEHAVIOR: The participant will have at least two months experience performing any kind of chemical analyses in a laboratory.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will use common laboratory safety equipment (e.g., glasses, pipeting devices, laboratory aprons) in performing all laboratory excercises, and will avoid eating and/or smoking in the laboratory.

B. Conditions:

He/she will have heard the lecture on the Subject Matter.

C. Accepted Performance:

The instructor will judge whether or not the participant used the common laboratory safety equipment and did not smoke or eat in the laboratory.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the Training Manual: Laboratory Safety Practices.

2. X-27, Laboratory Safety Practices (40 slides); see XI, Description of Visual Materials (page 9-3).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.

2. Check all laboratory safety devices for proper functioning, e.g., fume hoods, eye-wash fountains, fire extinguishers, etc.

B. Sequencing:

1. Classroom - discuss safety using X-27, Laboratory Safety Practices.

   a. Lab safety generally falls under the jurisdication of one of these two; X-27, 1.

   b. At one time, USEPA considered withholding certification of drinking water analytical labs which had persistent, major safety problems; X-27, 2.

   c. A good lab safety program requires three elements; X-27, 3, 4, and 5.

   d. Example chain of command through which safety regulations are promulgated in a company. Chance for confusion is high because of all the levels; X-27, 6.

   e. Introducing the next six slides which show possible lab accidents; X-27, 7.

   f. Possible lab accidents; X-27, 8 through 12, and 13 (the unforseen hazard).

   g. Introducing the next eleven slides which are photos of common lab safety errors; X-27, 14.

   h. Common lab safety errors; X-27, 15 through 22, 23 (possible back-siphon), 24 (can overflowing and not marked glass or paper), and 25 (no emergency phone numbers).

   i. Introducing the next thirteen slides which show common lab safety equipment; X-27, 26.

   j. Common lab safety equipment; X-27, 27 through 29 and 30, 31, 32 (first aid kits are not a substitute for professional help), 33 (types A, B, and C), 34 (siphon breaker), and 35 through 39.

   k. Purpose of safety program is accident prevention, not to be able to call an ambulance after the accident has occurred; X-27, 40.

2. Laboratory - point out the location of such safety devices as the fume hoods, eye-wash fountains, fire extinguishers, etc.

3. Laboratory - for each laboratory exercise, point out any special safety problems; e.g., evaporation of a flammable solvent, use of toxic chemicals, handling of acid solutions, etc.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

As specified for each laboratory exercise.

X. IPW REAGENT REQUIREMENTS:

As specified for each laboratory exercise.

XI. DESCRIPTION OF VISUAL MATERIALS

SERIES X-27, LABORATORY SAFETY PRACTICES

PRINTING:

STATE/SAFETY HEALTH PROGRAM

OCCUPATIONAL SAFETY & HEALTH ACT

SLIDE 1

CARTOON OF SAFETY CERTIFICATE BEING DENIED BECAUSE OF HAZARDOUS LABORATORY PRACTICE

SLIDE 2

PRINTING:

SAFETY REQUIRES COMMON SENSE

SLIDE 3

PRINTING:

SAFETY COSTS MONEY

SLIDE 4

PRINTING:

SAFETY REQUIRES CHANGES IN ATTITUDE

SLIDE 5

PRINTING:

TOP MANAGEMENT → MIDDLE MANAGEMENT → PLANT SUPERVISOR → FOREMAN → PLANT WORKER

SLIDE 6

PRINTING:

LIFE-HEALTH ENDANGERING LABORATORY ACCIDENTS

SLIDE 7

CARTOON OF DEAD LAB WORKER WITH SANDWICH IN HAND AND A BOTTLE MARKED "TOXIC" NEARBY

SLIDE 8

CARTOON OF UNCONSCIOUS LAB WORKER WITH UNSTOPPERED BOTTLE MARKED "TOXIC-VOLATILE" NEARBY

SLIDE 9

SERIES X-27, LABORATORY SAFETY PRACTICES

CARTOON OF OPEN FLAMES ON A LAB TABLE WITH OTHER ITEMS (INCLUDING A WORKER'S LAB COAT) ON FIRE

SLIDE 10

DRAWING OF ACID DRIPPING FROM A TIPPED FLASK ONTO A LAB WORKER'S CLOTHING AND THE FLOOR

SLIDE 11

DRAWING OF A LAB WORKER WITH LIGHTED CIGARETTE WORKING NEXT TO A BOTTLE OF LIQUID MARKED "FLAMMABLE"

SLIDE 12

CARTOON DEPICTING HAZARDS OF PUBLIC RELATIONS - LAB WORKER BEING HIT WITH A BASEBALL BAT BY A WOMAN WHO IS SAYING, "MY WATER TASTES TERRIBLE."

SLIDE 13

PRINTING:

LABORATORY BOO-BOOS

SLIDE 14

PHOTO OF CLUTTERED WORK AREA

SLIDE 15

PHOTO OF LAB WORKER MOUTH-PIPETTING

SLIDE 16

PHOTO OF LAB WORKER POURING SULFURIC ACID WITH NONE OF THE USUAL PRECAUTIONS

SLIDE 17

PHOTO OF LAB WORKER CARRYING BOTTLE OF SULFURIC ACID WITHOUT A SAFETY CARRIER, ETC.

SLIDE 18

SERIES X-27 LABORATORY SAFETY PRACTICES

PHOTO OF LAB WORKER SMOKING A CIGARETTE WHILE TAKING A READING FROM A BURET

SLIDE 19

PHOTO OF LAB WORKER EATING AN APPLE WHILE WATCHING A BURET DURING A TITRATION

SLIDE 20

PHOTO OF LAB WORKER DRINKING FROM A BEAKER AT A LAB BENCH

SLIDE 21

PHOTO OF A BOTTLE OF "HEXANES" WITH CAP OFF, OUTSIDE A HOOD

SLIDE 22

PHOTO OF HOSE FROM A SINK FAUCET IMMERSED IN THE SINK

SLIDE 23

PHOTO OF A FILLED WASTE CAN WITH NO MARKING ABOUT GLASS OR PAPER, ETC.

SLIDE 24

PHOTO OF PHONE IN A LAB BUT NO EMERGENCY NUMBERS ARE POSTED

SLIDE 25

PRINTING:
COMMON SAFETY EQUIPMENT

SLIDE 26

PHOTO OF PIPET BULBS

SLIDE 27

SERIES X-27, LABORATORY SAFETY PRACTICES

PHOTO OF TWO PIPETORS

SLIDE 28

PHOTO OF LAB WORKER WEARING PLASTIC APRON

SLIDE 29

PHOTO OF PAIRS OF SAFETY GLASSES

SLIDE 30

PHOTO OF VOLATILE, FLAMMABLE CHEMICAL PROPERLY STORED IN A HOOD

SLIDE 31

PHOTO OF A FIRST AID KIT

SLIDE 32

PHOTO OF A FIRE EXTINGUISHER WITH AN INSPECTION TAG

SLIDE 33

PHOTO OF SINK AREA WITH NO GLASSWARE STANDING IN OR AROUND IT. THE HOSE FROM THE FAUCET COMES JUST TO THE TOP OF THE SINK.

SLIDE 34

PHOTO OF ACID BOTTLES IN TWO TYPES OF SAFETY CONTAINERS

SLIDE 35

PHOTO OF AN EXPLOSION-PROOF REFRIGERATOR

SLIDE 36

PHOTO TAKEN CLOSE-UP OF THE EXPLOSION-PROOF LABEL ON A REFRIGERATOR

SLIDE 37

PHOTO OF SIGN ON A REFRIGERATOR, "DO NOT STORE HAZARDOUS MATERIALS IN THIS UNIT".

SLIDE 38

PHOTO OF SIGNS ON A REFRIGERATOR, "DO NOT STORE FLAMMABLE MATERIALS IN THIS UNIT."

SLIDE 39

DRAWING OF AMBULANCE LEAVING TREATMENT PLANT

SLIDE 40 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Dissolved Oxygen (DO) - Winkler
Dissolved Oxygen (DO) - Meter
Biochemical Oxygen Demand (BOD)

II. UNIT OF INSTRUCTION: Summary of Topic Presentation (Although the subjects shown above are three distinct topics on the suggested agenda, they are covered as one item in this IPW because of their close interrelationship.)

III. ESTIMATED TIME: 5 hours, 10 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for these determinations to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of high school chemistry or equivalent, and have the ability to:

A. Perform weighings on a trip balance.

B. Use volumetric and graduated pipets, with pipet bulb.

C. Perform a titration.

D. Handle solutions of acids and bases safely.

E. Perform simple calculations (addition, subtraction, multiplication, division) involving decimals; convert percentages to decimal equivalents.

F. Use ordinary laboratory glassware; beakers, flasks, graduated cylinders, siphons, graduated pipets, volumetric pipets, etc.

G. Understand the terms µg, mg, g, ml, and l as being metric units of mass and volume.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on the Subject Matter, will use good laboratory technique in obtaining reasonable answers for the two DO determinations, and will obtain a BOD result using the DO values and % of sample dilution. The two DO results may be obtained using the Winkler procedure or a meter. In this IPW however, only the Winkler titration method is covered.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual, and the equipment/supplies/reagents listed in sections IX and X of this IPW.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. Judgement of the instructor regarding the laboratory technique.

3. Judgement of the instructor regarding the two DO values.

4. Correct BOD result as determined by using the formula for calculation of BOD.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Six outlines in the Training Manual:

   a. Dissolved Oxygen, Factors Affecting DO Concentration in Water

   b. Dissolved Oxygen, Determination by the Winkler Iodometric Titration - Azide Modification

   c. Dissolved Oxygen, Determination by Electronic Measurement

   d. Biochemical Oxygen Demand Test Procedures

   e. Laboratory Procedure for Dissolved Oxygen, Winkler Method - Azide Modification (According to Methods for Chemical Analysis of Water and Wastes, US EPA, Environmental Monitoring and Support Laboratory, 1974)

   f. Biochemical Oxygen Demand Test Dilution Technique (According to Standard Methods for the Examination of Water and Wastewater, 14 edition, page 543, 1975)

   Note: These outlines contain far more information than is needed by the students during the course. Much of the information is therefore "back-up", in nature.

2. X-31, Dissolved Oxygen - General Considerations (7 slides); See XI, Description of Visual Materials (page 10-14).

3. XT-29, Dissolved Oxygen Determination (72 slides, 15 min. tape, script); See XI, Description of Visual Materials (page 10-15 ff).

4. X-32, YSI DO Meter (29 slides); See XI, Description of Visual Materials (page 10-23 ff).

5. XT-54, The Determination of Biochemical Oxygen Demand (71 slides, 12 min. tape, script); See XI, Description of Visual Materials (page 10-27 ff).

B. Suggested Media:

XT or X unit on the standardization of sodium thiosulfate.

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
2. Review the laboratory procedure by performing the student assignment.
3. Reproduce the attached quiz; one copy for each participant, plus any desired file copies. See pages 10-9 through 10-13.
4. Prepare the equipment/supplies/reagents/ listed in Sections IX and X of this IPW.
5. Obtain a sample of municipal wastewater treatment plant effluent. The amount of sample at each laboratory station will depend on the % dilution to be made by the participants for the BOD test. (A maximum of 300 ml per laboratory station will be needed.) Make sure the sample is unchlorinated, and that its pH is near 7. Otherwise, Sections III A. and III B. in the BOD outline (VII A. 1. f.) must be done by the instructor prior to the start of the laboratory.

B. Sequencing:

1. Classroom - discuss DO, using X-31, Dissolved Oxygen - General Considerations.
   a. Introduces the three topics to be covered. (The next six slides are part of the Genral Considerations) X-31, 1.
   b. Five reasons why the DO test is important. (The first is the biological "health" of a body of water; low DO, poor "health". The second refers to using the DO test to determine what DO conditions exist in the various units of a treatment plant. DO used to determine if boiler water might be corrosive because of DO content. DO used to calculate aeration costs). X-31, 2.
   c. Normal DO content of water; X-31, 3.
   d. Factors contributing to amount of DO in water; X-31, 4.
   e. DO value is a net observation of the factors cited above; X-31, 5.
   f. In considering water quality, minimum DO values are important, not average values; X-31, 6.
   g. Overall, DO is a measure of water quality; X-31, 7.
2. Classroom - discuss the Winkler procedure azide modification using XT-29, Dissolved Oxygen Determination.
   a. Introduction; XT-29, 1-3.
   b. Topics to be covered; XT-29, 4-9.

c. History of the test; XT-29, 10-13.

d. Sample collection, XT-29, 14-17.

e. Addition of reagents, and accompanying chemical reactions; XT-29, 18-33.

f. Interference in the determination; XT-29, 34-35.

g. Titration of the sample and accompanying chemical reaction; XT-29, 36-46.

h. Calculations in the determination; XT-29, 47-54.

i. Other interferences in the determination; XT-29, 55-64.

j. Preservation techniques; XT-29, 69-72.

k. Summary and credits, XT-29, 69-72.

3. Classroom - discuss the need to standardize the sodium thiosulfate. (When item VII B. becomes available, use it for this discussion.)

4. Classroom - discuss dissolved oxygen meters using X-32, YSI DO meter.

a. Usefulness of DO meters; X-32, 1-2.

b. General types of DO meters; X-32, 3.

c. Interferences. (Dissolved inorganic salts affect oxygen solubility, but are not really an interference in the use of the meter itself. Chlorine and hydrogen sulfide can corrode the electrodes on prolonged exposure). X-32, 4.

d. Items to be aware of in using the meter. (Most meters for field use are pressure compensated). X-32, 5.

e. Methods (NPDES approved, and other) of meter claibration. "Saturated" refers to saturating water with oxygen, checking its temperature, and reading the DO from a table). X-32, 6.

f. Photo of field probe without protective end; X-32, 7.

g. Photo of field probe with protective end on; X-32, 8.

h. Photo of end of BOD bottle - model probe: X-32, 9.

i. Photo of top of BOD bottle - model probe; X-32, 10.

j. Photo of electrical jacks on the side of the meter; X-32, 11.

k. Photo of meter face, with nad on selector switch, and BOD bottle - model probe in BOD bottle; X-32, 12.

l. Photo of membrane o-ring being removed; X-32, 13.

   m. Photo of electrolyte being prepared; X-32, 14.

   n. Photo of electrolyte being transferred to eyedropper bottle; X-32, 15.

   o. Photo of membrane packet; X-32, 16.

   p. Photo of membrane being placed on BOD bottle - model probe; X-32, 17.

   q. Photo of electrolyte being placed in end of BOD bottle - model probe; X-32, 18.

   r. Photo of membrane being drawn over end of BOD bottle - model probe; X-32, 19.

   s. Photo of o-ring being placed over end of probe; X-32, 20.

   t. Photo of membrane in place on BOD bottle - model probe; X-32, 21.

   u. Photo of excess membrane being cut away; X-32, 22.

   v. Photo of meter, with probe being placed in BOD bottle; X-32, 23.

   w. Photo of needle being mechanically adjusted to zero; X-32, 24.

   x. Photo of meter selector switch being turned to red-line position; X-32, 25.

   y. Photo of red-line adjustment being made; X-32, 26.

   z. Photo of zero adjustment being made; X-32, 27.

   aa. Photo of calibration adjustment being made; X-32, 28.

   bb. Photo of meter being turned off; X-32, 29.

5. Classroom - discuss BOD using XT-54, The Determination of Biochemical Oxygen Demand.

   a. Introduction; XT-54, 1-2.

   b. Topics to be covered; XT-54, 3-8.

   c. Sources of the procedure; XT-54, 9-10.

   d. Usefulness of the test; XT-54, 11-12.

   e. Description of the test; XT-54, 13-14.

   f. Preparation of dilution water; XT-54, 15-30.

   g. Preparation of nutrient salts; XT-54, 31-33.

   h. Seed materials; XT-54, 34-40.

   i. Sample pretreatment; XT-54, 41-44.

j. Treatment of samples saturated with oxygen; XT-54, 45-47.

k. Sample dilution and storage; XT-54, 48-57.

l. Determination of dissolved oxygen in the diluted sample; XT-54, 58-60.

m. Calculation of BOD result; XT-54, 61-62.

n. Determination of unseeded blank; XT-54, 63-66.

o. Dissolved oxygen meter as a substitute for the Winkler procedure; XT-54, 67.

p. Summary and credits; XT-54, 68-70.

6. Classroom - inform the participants what dilutions they will use in performing the BOD test. The amount of sample used must be determined by the instructor using previous experience or information from other sources. Recall that during the usual five day incubation period, at least 2 mg of DO/l must be consumed, and at least 1 mg of DO/l must remain.

7. Classroom - inform the participants that in a 5 day course, a 4 day BOD will be done, (i.e., Monday-Friday), even though 5 days is the ordinary incubation period.

8. Laboratory - have the participants do section III of the Training Manual outline, Laboratory Procedure for Dissolved Oxygen, Winkler Method - Azide Modification.

9. Laboratory - have the participants do section V of the Training Manual outline Biochemical Oxygen Demand Test Dilution Technique.

10. Laboratory - have the participants do section IV of the outline cited in 8.

11. Laboratory - last day of the incubating period. Have the participants do section IV of the outline cited in 9, using the second BOD bottle.

12. Classroom - have the participants do section VI of the outline cited in 9.

13. Classroom - final discussion and questions.

14. Classroom - give the quiz and then read the correct answers from the attached answer sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 1 sample

A. For Each Station:

1 pair safety glasses (for each person at the station)

1 laboratory apron or coat (for each person at the station)

1 distilled water plastic squeeze bottle

paper towels or sponge

1 grease pencil (or other marking pencil)

1 1 or 2 inch square plastic weighing boat

1 1 liter graduated cylinder

1 100 ml graduated cylinder

1 10 ml graduated cylinder

1 500 ml Erlenmeyer flask

2 300 ml ± 3 ml BOD bottles

1 siphon; long enough for use with 1 liter graduated cylinder

1 plunger-type mixer; if not available, a long stirring rod (for use with the 1 liter graduated cylinder) may be substituted

1 25 ml buret

1 buret clamp

1 ring stand

1 small funnel (to fit into the buret)

1 pipet bulb

1 20 ml volumetric pipet

1 10 ml graduated pipet

3 2 ml volumetric or graduated pipets

B. Shared:

Trip balance, 1 per 4 stations

Scoop-type spatula, 1 at each balance

4 1 ml volmetric pipets, 1 set per 4 stations

X. IPW REAGENT REQUIREMENTS:

Minimum Amounts for Each Station
(Prepare excess reagent to allow for waste, rinsing of glassware, and any desired re-runs).

Instructions for preparing the reagents may be found in section II of the Training Manual outline Laboratory Procedure for Dissolved Oxygen, Winkler Method - Azide Modification, and in section II of the outline Biochemical Oxygen Demand Test Dilution Technique. Reagents II H, II I, II J, II K, II L and 2 N in the BOD outline will not be needed if the pH sample is near 7, and if it is unchlorinated. Reagent II M is unnecessary if reagent II D was prepared as part of the DO outline. Either may be used.

3 g	potassium iodide
10 ml	10% sulfuric acid
45 ml	approximately 0.0375 N sodium thiosulfate
20 ml	0.0375 N potassium biiodate
4 ml	manganous sulfate
4 ml	alkaline-iodide-azide
4 ml	concentrated sulfuric acid
4 ml	starch

The following reagents (1 ml per station) may be in "community" bottles (1 set of 4 bottles per 3 stations), with a 1 ml volumetric pipet in each bottle.

phosphate buffer

magnesium sulfate

calcium chloride

ferric chloride

NAME____________________________

QUIZ

Dissolved Oxygen (DO) - Winkler
Dissolved Oxygen (DO) - Meter
Biochemical Oxygen Demand (BOD)

Check the correct answer or fill in the missing word(s) as indicated.

1. The DO test is the basis of the BOD test.

   _____T

   _____F

2. At sea-level and 20°C, distilled water holds about 9.2 mg of DO/l.

   _____T

   _____F

3. One action which increases the amount of DO in water is ____________________.

4. One action which decreases the amount of DO in water is ____________________.

5. The normality of the sodium thiosulfate titrant must be exactly 0.0375 for use in the DO test.

   _____T

   _____F

6. Given the following information regarding the standardization of sodium thiosulfate;

   ml of potassium bi-iodate used = 20.0
   N of potassium bi-iodate = 0.0375
   ml of sodium thiosulfate used = 19.0

   What is the N of sodium thiosulfate?

   _____0.0375

   _____0.0395

   _____0.0365

7. In a DO titration, the endpoint is signaled by a color change of ____________________ to ____________________________.

8. In general, DO probes are subject to more interferences than the Winkler titration.

   _____T

   _____F

NAME________________________________

9. In most DO probes, the electrodes are separated from the water by a

___________________________________.

10. DO probes should never be used if the sample contains chlorine.

_____T

_____F

11. BOD is a direct measure of organic matter.

_____T

_____F

12. In the BOD test, the amount of oxygen in the incubated BOD bottle _____________ during the incubation period.

13. Which of the following give a response in the BOD test? (One point will be deducted for each wrong guess.)

______sodium hydroxide

______carbonaceous materials

______sodium sulfite

______metallic mercury

______nitrogeneous materials

14. The incubation temperature in the BOD test is:

_____25°C

_____15°C

_____20°C

15. Sample pH has no influence on a BOD result.

_____T

_____F

16. If a BOD sample has been dechlorinated, it must be _______________before using it in the test.

17. Given the following information, what is the BOD?

Initial DO = 7.6 mg/l

Final DO = 2.8 mg/l

ml of sample diluted to 1 l = 30

_____375 mg/l

_____160 mg/l

_____250 mg/l

NAME____________________

QUIZ - ANSWER SHEET

Dissolved Oxygen (DO) - Winkler
Dissolved Oxygen (DO) - Meter
Biochemical Oxygen Demand (BOD)

Check the correct answer or fill in the missing word(s) as indicated.

1. The DO test is the basis of the BOD test.

   _X_ T

   ___ F

2. At sea-level and 20°C, distilled water holds about 9.2 mg of DO/l.

   _X_ T

   ___ F

3. One action which increases the amount of DO in water is PHOTOSYNTHESIS OR AERATION.

4. One action which decreases the amount of DO in water is RESPIRATION OR NITRIFICATION.

5. The normality of the sodium thiosulfate titrant must be exactly 0.0375 for use in the DO test.

   ___ T

   _X_ F

6. Given the following information regarding the standardization of sodium thiosulfate,

   ml of potassium bi-iodate used = 20.0
   N of potassium bi-iodate = 0.0375
   ml of sodium thiosulfate used = 19.0

   What is the N of sodium thiosulfate?

   ___ 0.0375

   _X_ 0.0395

   ___ 0.0365

7. In a DO titration, the endpoint is signaled by a color change of BLUE OR PALE BLUE to COLORLESS.

8. In general, DO probes are subject to more interferences than the Winkler titration.

   ___ T

   _X_ F

NAME ____________________

9. In most DO probes, the electrodes are separated from the water by a MEMBRANE OR SEMI-PERMEABLE MEMBRANE.

10. DO probes should never be used if the sample contains chlorine.

___ T

_X_ F

11. BOD is a direct measure of organic matter.

___ T

_X_ F

12. In the BOD test, the amount of oxygen in the incubated BOD bottle DECREASES during the incubation period.

13. Which of the following give a response in the BOD test? (One point will be deducted for each wrong guess.)

___ sodium hydroxide
_X_ carbonaceous materials
_X_ sodium sulfite
___ metallic mercury
_X_ nitrogeneous materials

14. The incubation temperature in the BOD test is:

___ 25°C
___ 15°C
_X_ 20°C

15. Sample pH has no influence on a BOD result.

___ T

_X_ F

16. If a BOD sample has been dechlorinated, it must be RESEEDED before using it in the test.

17. Given the following information, what is the BOD?

Initial DO = 7.6 mg/l

Final DO = 2.8 mg/l

ml of sample diluted to 1 l = 30

___ 375 mg/l

_X_ 160 mg/l

___ 250 mg/l

Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For fill-in answers, any which are equivalent to those indicated above would, of course, also be correct.

XI. DESCRIPTION OF VISUAL MATERIALS

SERIES X-31, DISSOLVED OXYGEN-GENERAL CONSIDERATIONS

PRINTING:

THE DISSOLVED OXYGEN DETERMINATION

- GENERAL CONSIDERATIONS
- WET CHEMICAL METHODS (WINKLER-AZIDE)
- ELECTROCHEMICAL METHODS (METER, PROBE)

SLIDE 1

PRINTING:

IMPORTANCE OF THE DISSOLVED OXYGEN TEST

- BIOLOGICAL
- AEROBIC OR ANAEROBIC
- BASIS OF THE BOD TEST
- WATER CORROSIVENESS
- AERATION COSTS

SLIDE 2

PRINTING:

20°C

9.2 mg OF DO/LITER

SLIDE 3

PRINTING:

DISSOLVED OXYGEN
(DO BALANCE)

INCREASED BY	DECREASED BY
·PHOTOSYNTHESIS	·RESPIRATION
·AERATION	·NITRIFICATION

SLIDE 4

PRINTING:

NET EFFECTS ARE OBSERVED

SLIDE 5

PRINTING:

AQUATIC LIFE SURVIVES ON MINIMUM DO VALUES, NOT AVERAGE VALUES

SLIDE 6

PRINTING:

IN SUMMARY, A MEASURE OF WATER QUALITY

SLIDE 7 (last slide)

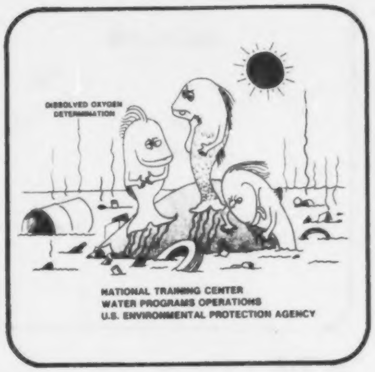
SLIDE

SLIDE

US EPA (LOGO)

PRESENTS

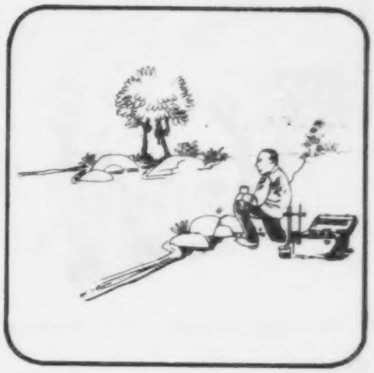
SLIDE 1



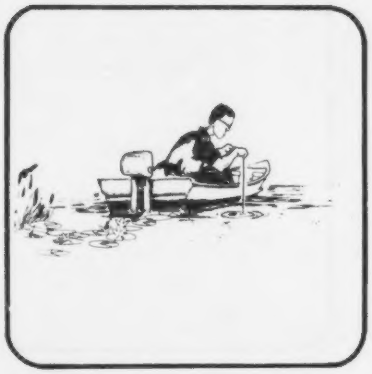
DISSOLVED OXYGEN DETERMINATION

NATIONAL TRAINING CENTER
WATER PROGRAMS OPERATIONS
U.S. ENVIRONMENTAL PROTECTION AGENCY

SLIDE 2



SLIDE 3



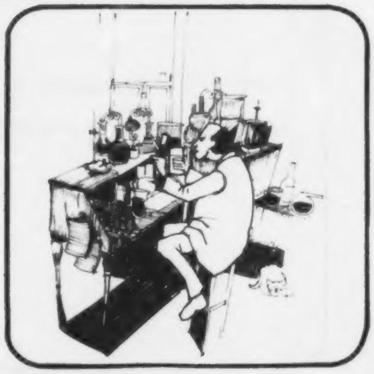
SLIDE 4

WINKLER DISSOLVED
OXYGEN DETERMINATION

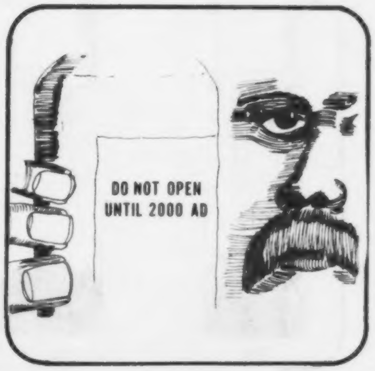
SLIDE 5

ALSTERBERG AZIDE MODIFICATION

SLIDE 6

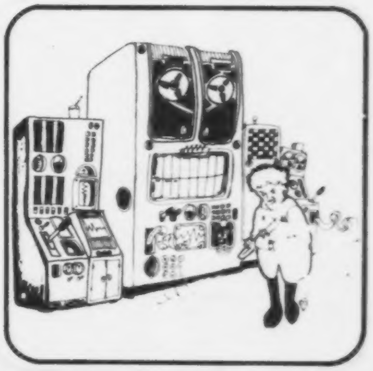


SLIDE 7



DO NOT OPEN
UNTIL 2000 AD

SLIDE 8



SLIDE 9

SERIES XT-29, DISSOLVED OXYGEN DETERMINATION

SLIDE 10

MODIFICATIONS

ALSTERBERG

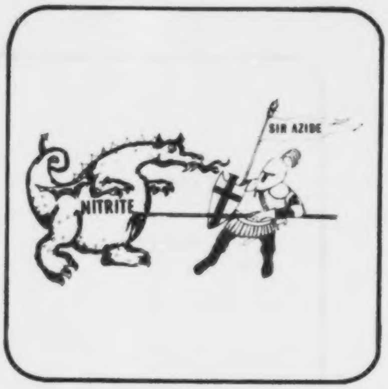
ALUM FLOCCULATION

SHORT THERIAULT

ALKALINE HYPOCHLORITE

RIDEAL — STEWART

SLIDE 11



SLIDE 12

PHOTO OF MANUAL COVER:

METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES

SLIDE 13

PHOTO OF

300 ml BOD BOTTLE

SLIDE 14

PHOTO OF

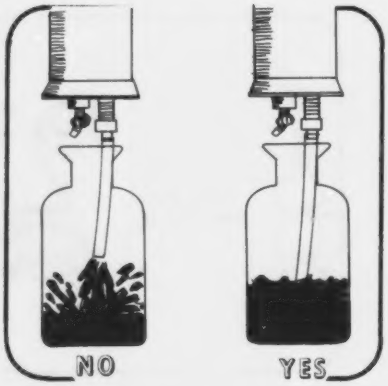
APHA SAMPLER

SLIDE 15

PHOTO OF

KEMMERER SAMPLER

SLIDE 16



SLIDE 17

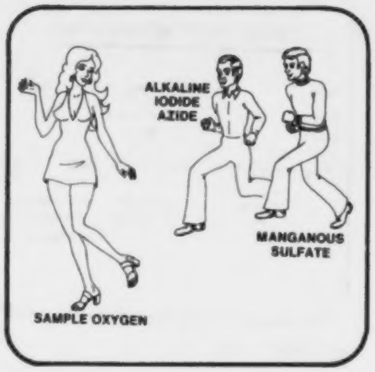
PHOTO OF 2 ml MANAGANOUS SULFATE BEING ADDED TO BOD BOTTLE FROM AUTO-PIPETOR

SLIDE 18

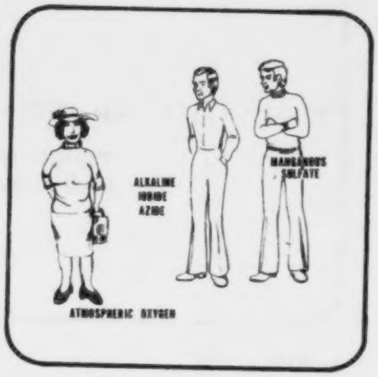
SERIES XT-29, DISSOLVED OXYGEN DETERMINATION

PHOTO OF 2 ml ALKALINE AZIDE IODIDE BEING ADDED TO BOD BOTTLE FROM AUTO-PIPETTOR.

SLIDE 19


ALKALINE
IODIDE
AZIDE
MANGANOUS
SULFATE
SAMPLE OXYGEN

SLIDE 20


MANGANOUS
SULFATE
ALKALINE
IODIDE
AZIDE
ATMOSPHERIC OXYGEN

SLIDE 21

PHOTO OF 2 BOD BOTTLES AFTER ADDITION OF 1st TWO DO REAGENTS. NO DO IN ONE BOTTLE. SOME DO IN THE 2nd.

SLIDE 22

PHOTO OF STOPPER BEING PLACED IN BOD BOTTLE. 1st TWO DO REAGENTS HAVE ALREADY BEEN ADDED.

SLIDE 23

PHOTO OF BOD BOTTLE BEING INVERTED TO MIX 1st TWO DO REAGENTS WITH SAMPLE.

SLIDE 24

PHOTO OF ALKALINE FLOC SETTLING IN BOD BOTTLE.

SLIDE 25

SAME AS 25, BUT ALSO SHOWING CLOCK.

SLIDE 26

$MnSO_4 + 2KOH \longrightarrow Mn(OH)_2 + K_2SO_4$

manganous sulfate

manganous hydroxide

SLIDE 27

SERIES XT-29, DISSOLVED OXYGEN DETERMINATION

$Mn(OH)_2 + \frac{1}{2}O_2 \longrightarrow MnO(OH)_2$

manganic hydroxide

SLIDE 28

ALSTERBERG AZIDE MODIFICATION

$NaN_3 + H^+ \longrightarrow HN_3 + Na^+$

sodium azide; hydrazoic acid

$HN_3 + NO_2^- + H^+ \longrightarrow N_2 + N_2O + H_2O$

nitrite ion; nitrogen (volatile); dinitrogen oxide (volatile)

SLIDE 29

PHOTO OF 2 ml SULFURIC ACID BEING ADDED TO BOD BOTTLE FROM AUTO-PIPETTOR.

SLIDE 30

PHOTO OF PACKET OF SULFAMIC ACID BEING ADDED TO BOD BOTTLE.

SLIDE 31

PHOTO OF BOD BOTTLE BEING INVERTED TO MIX ACID.

SLIDE 32

$MnO(OH)_2 + 2H_2SO_4 \longrightarrow Mn(SO_4)_2 + 3H_2O$

$Mn(SO_4)_2 + 2KI \longrightarrow MnSO_4 + K_2SO_4 + I_2$

red-brown

SLIDE 33

PHOTO OF BOD BOTTLE. CONTENTS ARE RED-BROWN IN COLOR. SOLID MATERIAL ON BOTTOM OF BOTTLE.

SLIDE 34

PHOTO OF FEW DROPS OF SULFURIC ACID BEING ADDED TO BOD BOTTLE.

SLIDE 35

PHOTO OF BOD BOTTLE BEING EMPTIED INTO WIDE MOUTH 500 ml ERLENMEYER FLASK.

SLIDE 36

SERIES XT-29, DISSOLVED OXYGEN DETERMINATION

PHOTO OF 0.0375 N SODIUM THIOSULFATE BEING ADDED TO 500 ml ERLENMEYER FLASK FROM BURET. SOLUTION COLOR IS RED-BROWN.

SLIDE 37

SAME AS 37, EXCEPT SOLUTION IS PALE YELLOW.

SLIDE 38

PRINTING:

PHENYLARSENE OXIDE

SLIDE 39

SAME AS 38, EXCEPT PHENLYARSENE OXIDE IS BEING ADDED.

SLIDE 40

PHOTO OF 2 ml OF STARCH BEING ADDED TO THE 500 ml ERLENMEYER FLASK.

SLIDE 41

PHOTO OF 500 ml ERLENMEYER FLASK BEING SWIRLED. SOLUTION COLOR IS DARK BLUE.

SLIDE 42

POWDERED STARCH INDICATOR

SLIDE 43

PRINTING:

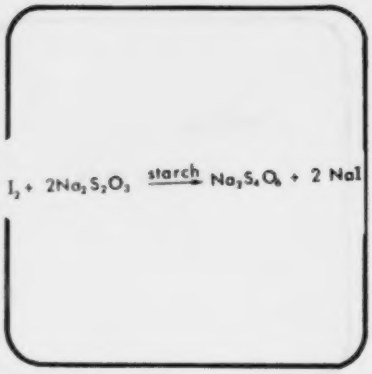
SAME AS 37, EXCEPT SOLUTION IS COLORLESS.

SLIDE 44

PHOTO OF BURET READING BEING RECORDED.

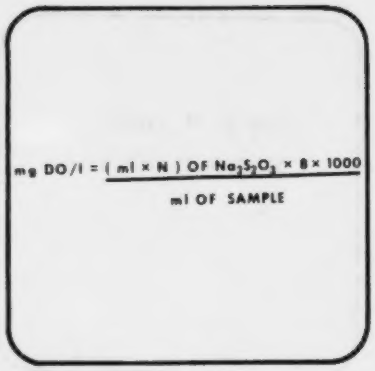
SLIDE 45

SERIES XT-29, DISSOLVED OXYGEN DETERMINATION



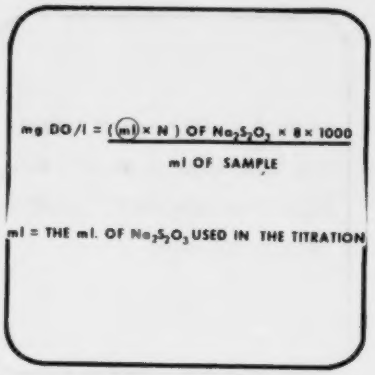
$I_2 + 2Na_2S_2O_3 \xrightarrow{starch} Na_2S_4O_6 + 2\,NaI$

SLIDE 46



$$\text{mg DO/l} = \frac{(\text{ml} \times \text{N}) \text{ OF } Na_2S_2O_3 \times 8 \times 1000}{\text{ml OF SAMPLE}}$$

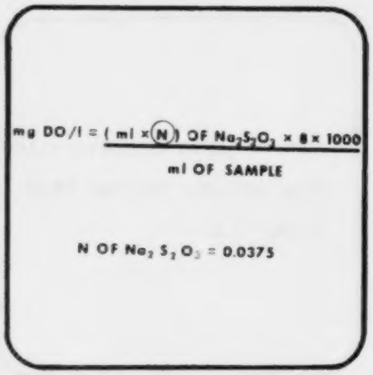
SLIDE 47



$$\text{mg DO/l} = \frac{(\boxed{\text{ml}} \times \text{N}) \text{ OF } Na_2S_2O_3 \times 8 \times 1000}{\text{ml OF SAMPLE}}$$

ml = THE ml. OF $Na_2S_2O_3$ USED IN THE TITRATION

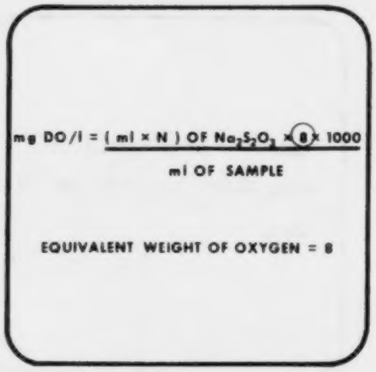
SLIDE 48



$$\text{mg DO/l} = \frac{(\text{ml} \times \boxed{\text{N}}) \text{ OF } Na_2S_2O_3 \times 8 \times 1000}{\text{ml OF SAMPLE}}$$

N OF $Na_2S_2O_3$ = 0.0375

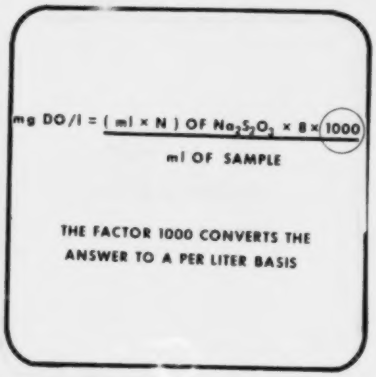
SLIDE 49



$$\text{mg DO/l} = \frac{(\text{ml} \times \text{N}) \text{ OF } Na_2S_2O_3 \times \boxed{8} \times 1000}{\text{ml OF SAMPLE}}$$

EQUIVALENT WEIGHT OF OXYGEN = 8

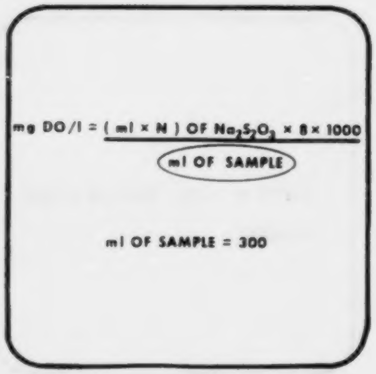
SLIDE 50



$$\text{mg DO/l} = \frac{(\text{ml} \times \text{N}) \text{ OF } Na_2S_2O_3 \times 8 \times \boxed{1000}}{\text{ml OF SAMPLE}}$$

THE FACTOR 1000 CONVERTS THE ANSWER TO A PER LITER BASIS

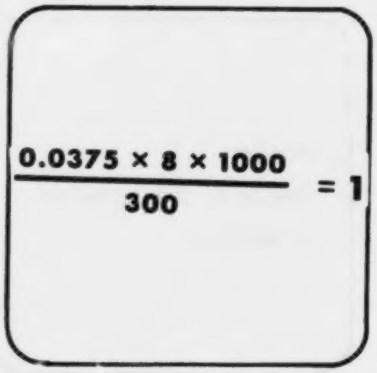
SLIDE 51



$$\text{mg DO/l} = \frac{(\text{ml} \times \text{N}) \text{ OF } Na_2S_2O_3 \times 8 \times 1000}{\boxed{\text{ml OF SAMPLE}}}$$

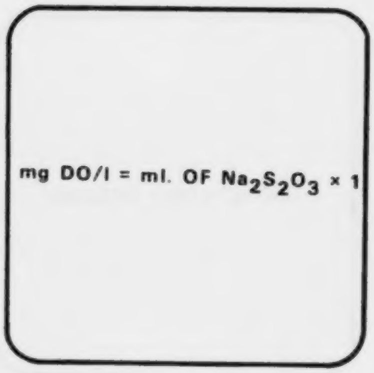
ml OF SAMPLE = 300

SLIDE 52



$$\frac{0.0375 \times 8 \times 1000}{300} = 1$$

SLIDE 53



mg DO/l = ml. OF $Na_2S_2O_3 \times 1$

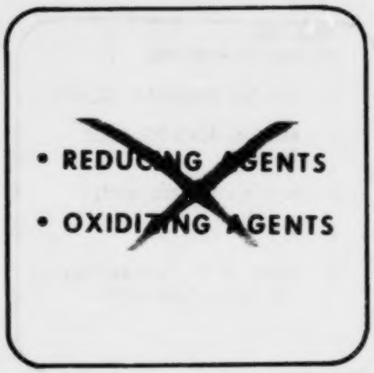
SLIDE 54

PRINTING:

·NITRITE NITROGEN

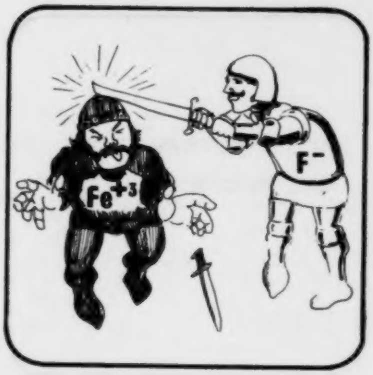
·LESS THAN 1 mg OF
FERROUS IRON PER LITER

SLIDE 55



- **REDUCING AGENTS**
- **OXIDIZING AGENTS**

SLIDE 56



SLIDE 57

PRINTING:

SULFITE

THIOSULFATE

POLYTHIONATE

FREE CHLORINE

SLIDE 58

PRINTING:

SUSPENDED SOLIDS

SLIDE 59

PRINTING:

ORGANICS OXIDIZED IN
AN ALKALINE MEDIUM OR
BY IODINE IN AN ACID
MEDIUM

SLIDE 60

PRINTING:

DOMESTIC SEWAGE

SLIDE 61

PRINTING:

BIOLOGICAL FLOCS

SLIDE 62

PRINTING:

COLORED SAMPLES

SLIDE 63

PHOTO OF WESTON AND STACK

MODEL 300 DO METER.

SLIDE 64

PRINTING:

PRESERVATION METHOD 1

1. ADD THE MANGANOUS SULFATE
2. ADD THE ALKALINE AZIDE IODIDE
3. STOPPER THE BOD BOTTLE
4. MIX THE CONTENTS
5. STORE AT THE TEMPERATURE OF THE COLLECTION WATER

SLIDE 65

PRINTING:

PRESERVATION METHOD 2

1. WATER SEAL THE BOTTLE
2. STORE IN THE DARK AT 10 - 20°C

SLIDE 66

PRINTING:

PRESERVATION METHOD 3

1. ADD 0.7 ml OF CONC. H_2SO_4
2. ADD 10 ml OF SODIUM AZIDE
3. STORE THE SAMPLES AS IN METHOD 1
4. COMPLETE THE PROCEDURE BY ADDING 2 ml OF MANGANOUS SULFATE, 3 ml OF ALKALI IODIDE, AND 2 ml OF CONC. H_2SO_4

SLIDE 67

PRINTING:

4 - 8 HOURS

MAXIMUM HOLDING TIME

SLIDE 68

PHOTO - SAME AS 13.

SLIDE 69

- ▲ SAMPLING TECHNIQUES
- ▲ BASIC CHEMISTRY OF THE WINKLER PROCEDURE
- ▲ BASIC CHEMISTRY OF THE ALSTERBERG AZIDE MODIFICATION
- ▲ REAGENT ADDITION
- ▲ SAMPLE PRESERVATION
- ▲ BASIC CALCULATIONS

SLIDE 70

PRINTING:

TECHNICAL REVIEW

P. F. HALLBACH

A. E. DONAHUE

C. R. FELDMANN

GRAPHICS COORDINATOR

M. D. STREFF

B. G. KRAMIG

NARRATION

C. R. FELDMANN

SLIDE 71

PRINTING:

CLEAN

WATER

SLIDE 72(LAST SLIDE)

XI. DESCRIPTION OF VISUAL MATERIALS

SERIES X-32, YSI DO METER

PRINTING:

DISSOLVED OXYGEN PROBES - RECOMMENDED IN CASES WHERE AN INTERFERENCE TO THE WINKLER METHOD EXISTS

SLIDE 1

PRINTING:

PROBE DETERMINATION IS

- SUITABLE FOR MONITORING
- NON-DESTRUCTIVE

SLIDE 2

PRINTING:

TYPES OF DISSOLVED OXYGEN PROBES

- GALVANIC
- POLAROGRAPHIC

SLIDE 3

PRINTING:

INTERFERENCES -

- NONE FROM ORGANIC MATERIALS
- DISSOLVED INORGANIC SALTS
- CHLORINE *
- HYDROGEN SULFIDE*

*Causes electrode corrosion on prolonged Exposure

SLIDE 4

PRINTING:

OTHER PROBLEMS -

MUST BE CALIBRATED

MUST BE CARED FOR

TEMPERATURE AND PRESSURE SENSITIVE

MEMBRANE (TEARING, PUNCTURE, GREASE AND OILS)

INSUFFICIENT FLOW ACROSS MEMBRANE

SLIDE 5

PRINTING:

DISSOLVED OXYGEN METER CALIBRATION

- IN AIR
- IN WATER
  - WINKLER
  - SATURATED

SLIDE 6

PHOTO OF END OF FIELD PROBE SHOWING MEMBRANE; PROTECTIVE END IS OFF.

SLIDE 7

PHOTO OF FIELD PROBE; PROTECTIVE END IS ON.

SLIDE 8

PHOTO OF END OF BOD BOTTLE - MODEL PROBE.

SLIDE 9

SERIES X-32, YSI DO METER

PHOTO OF TOP OF BOD BOTTLE - MODEL PROBE

SLIDE 10

PHOTO OF ELECTRICAL JACKS ON THE SIDE OF THE METER.

SLIDE 11

PHOTO OF THE METER FACE, WITH HAND ON THE SELECTOR SWITCH, AND BOD BOTTLE - MODEL PROBE IN BOD BOTTLE.

SLIDE 12

PHOTO OF MEMBRANE O-RING BEING REMOVED.

SLIDE 13

PHOTO OF ELECTROLYTE BEING PREPARED.

SLIDE 14

PHOTO OF ELECTROLYTE BEING TRANSFERRED TO EYEDROPPER BOTTLE.

SLIDE 15

PHOTO OF MEMBRANE PACKET.

SLIDE 16

PHOTO OF MEMBRANE BEING PLACED ON BOD BOTTLE - MODEL PROBE.

SLIDE 17

PHOTO OF ELECTROLYTE BEING PLACED IN END OF BOD BOTTLE - MODEL PROBE.

SLIDE 18

SERIES X-32, YSI DO METER

PHOTO OF MEMBRANE BEING DRAWN OVER END OF BOD BOTTLE - MODEL PROBE.

SLIDE 19

PHOTO OF O-RING BEING PLACED OVER END OF PROBE.

SLIDE 20

PHOTO OF MEMBRANE IN PLACE ON BOD BOTTLE - MODEL PROBE.

SLIDE 21

PHOTO OF EXCESS MEMBRANE BEING CUT AWAY.

SLIDE 22

PHOTO OF METER, WITH PROBE BEING PLACED IN BOD BOTTLE.

SLIDE 23

PHOTO OF NEEDLE BEING MECHANICALLY ADJUSTED TO ZERO.

SLIDE 24

PHOTO OF METER SELECTOR SWITCH BEING TURNED TO RED-LINE POSITION.

SLIDE 25

PHOTO OF RED-LINE ADJUSTMENT BEING MADE.

SLIDE 26

PHOTO OF ZERO ADJUSTMENT BEING MADE.

SLIDE 27

SERIES X-32, YSI DO METER

PHOTO OF CALIBRATION ADJUSTMENT BEING MADE.

SLIDE 28

PHOTO OF METER BEING TURNED OFF.

SLIDE 29 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

XI. DESCRIPTION OF VISUAL MATERIALS

SERIES XT-54, THE DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND

US EPA (LOGO)
PRESENTS

SLIDE 1

US ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF WATER PROGRAMS OPERATIONS
NATIONAL TRAINING & OPERATIONAL
TECHNOLOGY CENTER

SLIDE 2

PRINTING:

DETERMINATION
OF THE
BIOCHEMICAL OXYGEN DEMAND

SLIDE 3

PRINTING:

DILUTION WATER

SLIDE 4

PRINTING:

SEEDING

SLIDE 5

PRINTING:

SAMPLE PRETREATMENT

SLIDE 6

PRINTING:

SAMPLE DILUTION

SLIDE 7

PRINTING:

CALCULATIONS

SLIDE 8

PHOTO OF 14th EDITION OF
STANDARD METHODS

SLIDE 9

PHOTO OF 1974 EPA METHODS MANUAL

SLIDE 10

PRINTING:

OXYGEN REQUIREMENTS OF:

- MUNICIPAL WASTEWATERS
- INDUSTRIAL WASTEWATERS

SLIDE 11

PRINTING:

EFFECTS ON RECEIVING WATER

SLIDE 12

DRAWING OF A CALENDAR. ONE WEEK IS MARKED OFF.

SLIDE 13

CARTOON DEPICTING BACTERIA BREAKING DOWN ORGANIC MATTER.

SLIDE 14

PRINTING:

DILUTION WATER

SLIDE 15

PRINTING:

HIGH QUALITY DISTILLED WATER

SLIDE 16

PRINTING:

- BLOCK TIN
- ALL GLASS

SLIDE 17

LESS THAN 0.01 mg Cu/l

SLIDE 18

SERIES XT-54, THE DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND

PRINTING:

- CHLORINE
- CHLORAMINES
- CAUSTIC ALKALINITY
- ORGANIC MATERIAL
- ACID

SLIDE 19

PHOTO OF CARBOY OF WATER WITH COTTON PLUG IN NECK.

SLIDE 20

DRAWING OF ANALYST AGITATING BOTTLE OF WATER.

SLIDE 21

PHOTO OF WATER IN CARBOY BEING AERATED.

SLIDE 22

DRAWING OF ANALYST TAKING TEMPERATURE OF WATER IN CARBOY.

SLIDE 23

PHOTO OF ANALYST ADDING NUTRIENTS AND BUFFER TO WATER IN CARBOY.

SLIDE 24

PRINTING:

DILUTION WATER = DISTILLED WATER+ BUFFER + NUTRIENT SALTS

SLIDE 25

PRINTING:

INCUBATOR STORAGE

- 20°C ± 1°
- DARK

SLIDE 26

PRINTING:

ADD BUFFER JUST BEFORE USE

SLIDE 27

SERIES XT-54, THE DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND

PHOTO OF BOTTLES OF CHEMICALS USED TO PREPARE BUFFER.

SLIDE 28

PRINTING:

PHOSPHATE BUFFER

-8.5g KH_2PO_4

-21.75g K_2HPO_4

-33.4g $Na_2HPO_4 \cdot 7H_2O$

-1.7g NH_4Cl

SLIDE 29

PRINTING:

pH 7.2

SLIDE 30

PRINTING:

MAGNESIUM SULFATE NUTRIENT

22.5g $MgSO_4$ - $7H_2O/l$

SLIDE 31

PRINTING:

CALCIUM CHLORIDE NUTRIENT

27.5 g $CaCl_2/l$

SLIDE 32

PRINTING:

FERRIC CHLORIDE NUTRIENT

0.25g $FeCl_3$ - $6H_2O/l$

SLIDE 33

SAME AS 14.

SLIDE 34

PHOTO OF BOTTLE OF SEED MATERIAL

SLIDE 35

PRINTING:

- SETTLED DOMESTIC SEWAGE
- 20°C
- 24 - 36 HOURS
- 1 - 2 ml/l

SLIDE 36

PRINTING:

SPECIALIZED SEED MATERIALS

SLIDE 37

PRINTING:

EFFLUENT OF BIOLOGICAL WASTE TREATMENT PROCESSES

SLIDE 38

PRINTING:

- RECEIVING WATER
- 2 - 5 MILES
- UNTREATED WASTE

SLIDE 39

PRINTING:

TYPE AND AMOUNT OF SEED

SLIDE 40

PRINTING:

NEUTRALIZATION OF ACIDITY OR ALKALINITY

SLIDE 41

PRINTING:

1 - 2 HOUR STANDING

SLIDE 42

PRINTING:

Na_2SO_3

SLIDE 43

PRINTING:

TOXIC MATERIALS

SLIDE 44

PHOTO OF ANALYST CHECKING TEMPERATURE OF WATER IN CARBOY.

SLIDE 45

SAME AS 21

SLIDE 46

CARTOON DEPICTING OIL FROM PUMP SQUIRTING OIL INTO ANALYST'S FACE.

SLIDE 47

PRINTING:

SAMPLE TYPE	%DILUTION
STRONG TRADE WASTE	0.1 - 1.0
RAW & SETTLED SEWAGE	1 - 5
OXIDIZED EFFLUENTS	5 - 25
POLLUTED RIVER WATERS	25 - 100

SLIDE 48

PHOTO OF ANALYST SIPHONING WATER FROM CARBOY INTO GRADUATED CYLINDER.

SLIDE 49

PHOTO OF ANALYST ADDING SAMPLE TO GRADUATED CYLINDER.

SLIDE 50

SAME AS 49

SLIDE 51

PHOTO OF ANALYST MIXING CONTENTS OF GRADUATED CYLINDER.

SLIDE 52

PHOTO OF ANALYST SIPHONING CONTENTS OF GRADUATED CYLINDER INTO BOD BOTTLE.

SLIDE 53

PHOTO OF ANALYST STOPPERING BOD BOTTLE.

SLIDE 54

SERIES XT-54, THE DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND

PHOTO OF ANALYST PLACING BOD BOTTLES INTO PAN.

SLIDE 55

PHOTO OF ANALYST FILLING FLARED TOP OF BOD BOTTLE WITH DISTILLED WATER.

SLIDE 56

PRINTING:

INCUBATION

- DARK
- 5 DAYS
- 20°C

SLIDE 57

WINKLER PROCEDURE-AZIDE MODIFICATION

DISSOLVED OXYGEN METER

SLIDE 58

PRINTING:

5 DAYS

SLIDE 59

PRINTING:

mg DO/l - mg DO/l = mg DO/l
(INITIAL) (5 DAYS)

SLIDE 60

PRINTING:

$$\text{mg BOD/l} = \frac{7.0 - 3.0}{0.1} = 40$$

SLIDE 61

PRINTING:

BOD_5

SLIDE 62

PRINTING:

UNSEEDED DILUTION WATER

SLIDE 63

SERIES XT-54, THE DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND

PRINTING:

- ·FILLING
- ·INCUBATION
- ·TITRATION

SLIDE 64

PRINTING:

0.2 mg/l

SLIDE 65

PRINTING:

CHECK ON DILUTION WATER

SLIDE 66

PHOTO OF WESTON AND STOCK MODEL 300 DO METER AND PROBE

SLIDE 67

PRINTING:

- ·REASONS FOR THE BOD TEST
- ·PREPARATION OF DILUTION WATER
- ·SEEDING TECHNIQUES
- ·SAMPLE PRETREATMENT
- ·SAMPLE DILUTION
- ·CALCULATIONS

SLIDE 68

PRINTING:

AUTHOR: C. R. FELDMANN

TECHNICAL REVIEW: D. G. BALLINGER
P. F. HALLBACH
F. J. LUDZACK

NARRATOR: C. R. FELDMANN

GRAPHICS: GRAPHIC ARTS, NERC

SLIDE 69

PRINTING:

CLEAN
WATER

SLIDE 70 (LAST SLIDE)

SLIDE

SLIDE

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Carbon Analyses

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 1 hour, 30 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of high school chemistry, or equivalent, and understand the terms µg, mg, g, ml, and l as being metric units of mass and volume.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on the Subject Matter and will participate in the discussion and laboratory demonstration.

B. Conditions:

He/she will have heard the lecture on the Subject Matter.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. Attentiveness during the lecture, as judged by the instructor.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the Training Manual: Total Carbon Analysis.

2. XT-59, Determination of Total Organic Carbon (62 slides, 13 minute tape, script); see XI, Description of Visual Materials (page 11-6, ff).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.

2. Review the laboratory procedure by performing the student assignment.

3. Reproduce the attached quiz; one copy for each participant, plus any desired file copies. See pages 11-4 and 11-5.

3. Prepare the equipment/supplies/reagents listed in section IX and X of this IPW.

4. Check the carbon analyzer for proper functioning. Note that the purpose of the demonstration is to show operation of the instrument, rather than to obtain a "perfect" calibration graph, or "correct" value on a sample.

5. Obtain a small amount (25 ml) of municipal wastewater treatment plant effluent.

B. Sequencing:

1. Classroom - discuss carbon analyses using XT-59, Determination of Total Organic Carbon. Note that although the XT unit deals mainly with total organic carbon analyses, inorganic and total carbon values are also of interest.

   a. Introduction; XT-59, 1-2.

   b. Topics to be covered; XT-59, 3.

   c. Source of the procedure; XT-59, 4.

   d. Historical information; XT-59, 5-13.

   e. Step in the use of the total carbon analyzer; XT-59, 14-28.

   f. Information dealing with sample injection; XT-59, 29-33.

   g. Sample collection and preservation; XT-59, 34-36.

   h. Preparation of standards; XT-59, 37

   i. Instrument start-up; XT-59, 38-43.

   j. Preparation of calibration graph; XT-59, 44.

   k. Analysis of sample; XT-59, 45-53.

   l. Other instrumentation; XT-59, 54-59.

   m. Summary and credits; XT-59, 60-62.

2. Laboratory -

   a. Point out the features of the carbon analyzer (e.g., temperature control, oxygen control, injection port, oven, recorder, detection system).

   b. Indicate need for reproducible injection technique.

   c. Point out problems caused by solids in the sample.

   d. Indicate need to avoid syringe contamination.

   e. Have the participants inject the standards and sample.

f. Prepare a composite calibration graph.

g. Obtain a total carbon value for the sample.

3. Classroom - final discussion and questions.

4. Classroom - give the quiz and then read the correct answers from the attached answer sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

These items will be used by the instructor in conducting the demonstration of the carbon analyzer.

1 50 or 100 ml syringe

Several tissues

One carbon analyzer. Although the Training Manual outline deals largely with the Beckman analyzer, any analyzer may be used.

X. IPW REAGENT REQUIREMENTS:

These items will be used by the instructor in conducting the demonstration of the carbon analyzer.

Small beaker of high quality distilled water (for rinsing the syringe)

5 carbon standards

2.0424 g of potassium acid phthalate dissolved in water and diluted to to 1 l = 1 mg C/ml.

This stock solution may be diluted, to obtain 5 standards with concentrations of 0-100 mg C/l.

NAME______________________________

QUIZ

Carbon Analyses

Check the correct answer or fill in the missing word(s) as indicated.

1. Three types of carbon which can be determined are____________________,
____________________, and ____________________.

2. Total organic carbon values are equal to chemical oxygen demand values.

   ______T

   ______F

3. The oxygen used in the analyzer need not be free of carbon.

   ______T

   ______F

4. A ________________ container should be used to collect the sample.

5. Solids can be removed from the sample by ____________________.

6. If only organic carbon is to be determined, what two things are done to the sample? ______________________ and __________________.

7. The main advantage of a carbon analysis over biochemical or chemical oxygen demand is _________________.

NAME______________________

QUIZ - ANSWER SHEET

Carbon Analyses

Check the correct answer or fill in the missing word(s) as indicated.

1. Three types of carbon which can be determined are total, organic, and inorganic.

2. Total organic carbon values are equal to chemical oxygen demand values.

    ___ T

    X F

3. The oxygen used in the analyzer need not be free of carbon.

    ___ T

    X F

4. A glass container should be used to collect the sample.

5. Solids can be removed from the sample by filtration.

6. If only organic carbon is to be determined, what two things are done to the sample? acidification, purging, or bubbling an inert gas through the sample.

7. The main advantage of a carbon analysis over biochemical or chemical oxygen demand is speed or time.

Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For "fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

XI. DESCRIPTION OF VISUAL MATERIALS

SERIES XT-59, DETERMINATION OF TOTAL ORGANIC CARBON

US EPA (LOGO)
PRESENTS

SLIDE 1

PRINTING:
THE DETERMINATION OF TOTAL
ORGANIC CARBON
US ENVIRONMENTAL PROTECTION
AGENCY

SLIDE 2

PRINTING:
USEFULLNESS OF THE TOTAL
ORGANIC CARBON DETERMINATION

SLIDE 3

PHOTO OF US EPA METHODS MANUAL

SLIDE 4

PRINTING:
·TWO TESTS
·ORGANIC POLLUTANTS

SLIDE 5

PRINTING:

BIOCHEMICAL OXYGEN DEMAND
BOD

SLIDE 6

CARTOON DEPICTING MICROORGANISMS
BREAKING DOWN ORGANIC MATTER

SLIDE 7

DRAWING OF ANALYST AND SUPERVISOR
AT LAB BENCH DISCUSSING LENGTH
OF THE ANALYSIS.

SLIDE 8

PRINTING:

CHEMICAL OXYGEN DEMAND
COD

SLIDE 9

PHOTO OF CHEMICAL OXYGEN DEMAND SAMPLES REFLUXING.

SLIDE 10

SAME AS 8

SLIDE 11

CARTOON DEPICTING TWO ANALYSTS ARGUING.

SLIDE 12

CARTOON DEPICTING WASTEWATER TREATMENT PLANT OPERATOR LOOKING FOR DATA.

SLIDE 13

SAME AS 8

SLIDE 14

PRINTING:

BECKMAN SINGLE CHANNEL
BECKMAN MODEL 915
DOHRMAN
OCEANOGRAPHIC INTERNATIONAL

SLIDE 15

CARTOON DEPICTING ANALYST USING COMPUTER TO GATHER DATA.

SLIDE 16

PRINTING:

1. SAMPLE PRETREATMENT
2. SAMPLE INJECTION
3. RECORDING OF THE RESULT

SLIDE 17

PHOTO OF HAND HOLDING SYRINGE IN FRONT OF BECKMAN SINGLE CHANNEL TOC INSTRUMENT.

SLIDE 18

PHOTO OF SAMPLE BEING INJECTED.

SLIDE 19

PHOTO OF OPEN COMBUSTION CHAMBER.

SLIDE 20

PHOTO OF CLOSED COMBUSTION CHAMBER.

SLIDE 21

PHOTO OF HAND POINTING TO TEMPERATURE GAGE.

SLIDE 22

PRINTING:

- CATALYST
- OXYGEN

SLIDE 23

PRINTING:

$C \rightarrow CO_2$

SLIDE 24

PHOTO OF HAND ON OXYGEN CYLINDER.

SLIDE 25

PHOTO OF ANALYST POINTING AT INFRARED PART OF TOC INSTRUMENT.

SLIDE 26

PHOTO OF ANALYST POINTING TO CHART RECORDER.

SLIDE 27

PHOTO OF ANALYST POINTING AT COOLING FAN.

SLIDE 28

PHOTO OF LABELED "T".

SLIDE 29

PHOTO OF ANALYST HOLDING SYRINGE.

SLIDE 30

PRINTING:

LARGE PARTICLES

SLIDE 31

PHOTO OF ANALYST HOLDING SPRING-LOADED SYRINGE.

SLIDE 32

PHOTO OF BLENDER

SLIDE 33

PHOTO OF ANALYST HOLDING GLASS BOTTLE AND CUBITAINER.

SLIDE 34

PRINTING:

- 4°C
- NO SUNLIGHT
- NO ATMOSPHERIC OXYGEN

SLIDE 35

PRINTING:

- 2 HOURS
- pH 2
- HCl
- H_2SO_4

SLIDE 36

PRINTING:

POTASSIUM HYDROGEN PHTHALATE

1 -100 mg C/l

SLIDE 37

PHOTO OF ANALYST ADJUSTING TEMPERATURE.

SLIDE 38

PHOTO OF ANALYST ADJUSTING GAS FLOW RATE.

SLIDE 39

PHOTO OF ANALYST ADJUSTING ZERO CONTROL.

SLIDE 40

PHOTO OF ANALYST ADJUSTING SENSITIVITY.

SLIDE 41

PRINTING:

BLANK

SLIDE 42

PRINTING:

DUPLICATE INJECTIONS

SLIDE 43

PHOTO OF CALIBRATION GRAPH

SLIDE 44

PHOTO OF pH METER AND SAMPLE

SLIDE 45

PHOTO OF SAMPLE BEING PURGED.

SLIDE 46

PRINTING:

- TOTAL ORGANIC CARBON
- DISSOLVED ORGANIC CARBON

SLIDE 47

PRINTING:

PLASTIC IS A NO - NO

SLIDE 48

PHOTO OF CALIBRATION GRAPH AND SAMPLE VALUE.

SLIDE 49

PHOTO OF US EPA METHODS MANUAL

SLIDE 50

PRINTING:

UNACIDIFIED SAMPLE
↓
ORGANIC CARBON
+
INORGANIC CARBON

SLIDE 51

PRINTING:

ACIDIFIED SAMPLE
↓
ORGANIC CARBON

SLIDE 52

PRINTING:

TOTAL CARBON - ORGANIC CARBON =
INORGANIC CARBON

SLIDE 53

PRINTING:

DUAL CHANNEL ANALYZERS

SLIDE 54

PRINTING:

INORGANIC CARBON

SLIDE 55

PRINTING:

- 175°C
- CARBONATE
- HYDROGEN CARBONATE

SLIDE 56

PRINTING:

- 950°C
- TOTAL CARBON

SLIDE 57

PRINTING:

175°C

INORGANIC CARBON

SLIDE 58

PRINTING:

TOTAL CARBON - INORGANIC CARBON =
ORGANIC CARBON

SLIDE 59

PRINTING:

- USEFULLNESS OF THE TEST
- RECOMMENDED METHOD OF ANALYSIS

SLIDE 60

PRINTING:

AUTHOR: C. R. FELDMANN

TECHNICAL REVIEW: R. L. BOOTH
P. F. HALLBACH
R. C. KRONER
L. B. LOBRING
C. MOENCH
R. T. WILLIAMS

NARRATION: C. R. FELDMANN
GRAPHICS: GRAPHIC ARTS
NERC, CINCINNATI

SLIDE 61

PRINTING:

CLEAN
WATER

SLIDE 62 (LAST SLIDE)

SLIDE ______

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Nitrogen Cycle

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 30 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant will have completed one year of college inorganic chemistry, or equivalent.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on all the nitrogen topics (given at the end of the nitrogen topics), and will know the forms of nitrogen in the atmosphere, basic reactions, forms of nitrogen which are measured and by what methods, and why nitrogen forms are measured.

B. Conditions:

He/she will have heard the lecture on the Subject Matter.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. Attentiveness during the lecture, as judged by the instructor.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the Training Manual:

a. Sources and Analysis of Organic Nitrogren

b. Ammonia, Nitrites, and Nitrates

c. Determination of Kjeldahl Nitrogen (Micro Apparatus-Nesslerization)

2. X-35, Kjeldahl Nitrogen (10 slides); see XI, Description of Visual Materials (pages 12-3 and 12-4).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
2. The quiz and answer sheet may be found in the IPW titled Discussion and Quiz - Nitrogen Topics. The same quiz is used for all the nitrogen topics, and is given at the end of all of the nitrogen topics.

B. Sequencing:

Classroom - discuss two outlines in Training Manual, Sources and Analysis of Organic Nitrogen; and Ammonia, Nitrites and Nitrates, using selected slides from X-35, Kjeldahl Nitrogen.

1. Forms of nitrogen: N_2, NO_2^-, NO_3^-, NH_3, N_2O_3, N_2O_5
2. Their occurrence; X-35, 8
    a. N_2 - 4/5 of atmosphere
    b. NO_3^- - $N_2 + O_3$ (electrical discharge)
    c. NO_2^- - from NO_3^- (bacterial reduction)
    d. NH_3 - from NO_2^- (bacterial reduction)
3. Types of nitrogen measured; X-35, 10
4. Why nitrogen is measured; X-35, 9
5. Summary

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

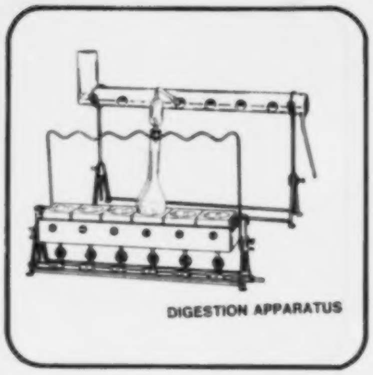
None

X. IPW REAGENT REQUIREMENTS:

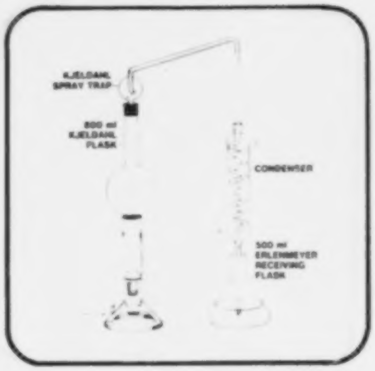
None

XI. DESCRIPTION OF VISUAL MATERIALS

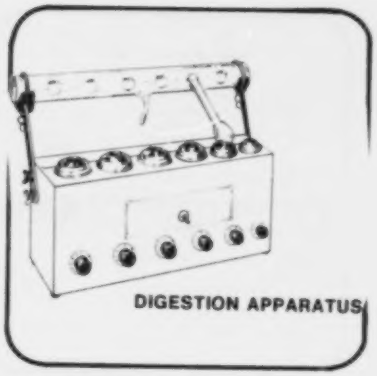
SERIES X-35, KJELDAHL NITROGEN


DIGESTION APPARATUS

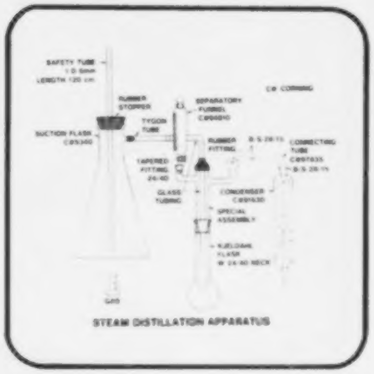
SLIDE 1


KJELDAHL SPRAY TRAP
800 ml KJELDAHL FLASK
CONDENSER
500 ml ERLENMEYER RECEIVING FLASK

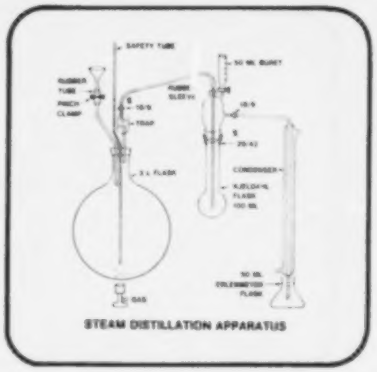
SLIDE 2


DIGESTION APPARATUS

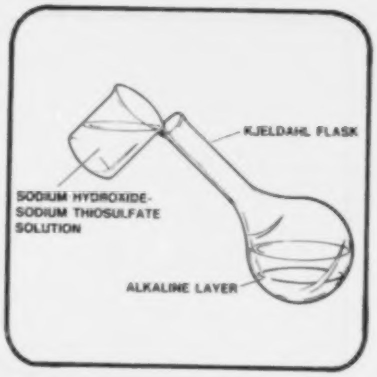
SLIDE 3


SAFETY TUBE
I.D. 6mm
LENGTH 120 cm
C# CORNING
RUBBER STOPPER
SEPARATORY FUNNEL
TYGON TUBE
SUCTION FLASK
RUBBER FITTING
CONNECTING TUBE
TAPERED FITTING 24/40
GLASS TUBING
CONDENSER
SPECIAL ASSEMBLY
KJELDAHL FLASK W 24/40 NECK
GAS
STEAM DISTILLATION APPARATUS

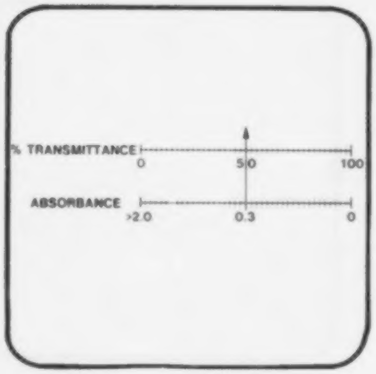
SLIDE 4


SAFETY TUBE
50 ML BURET
RUBBER TUBE
PINCH CLAMP
RUBBER SLEEVE
10/9
TRAP
10/9
20/42
3 L FLASK
CONDENSER
KJELDAHL FLASK 100 ML
50 ML ERLENMEYER FLASK
GAS
STEAM DISTILLATION APPARATUS

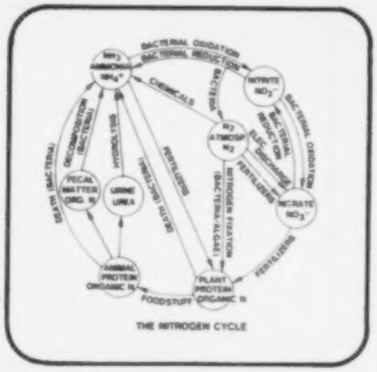
SLIDE 5


KJELDAHL FLASK
SODIUM HYDROXIDE-SODIUM THIOSULFATE SOLUTION
ALKALINE LAYER

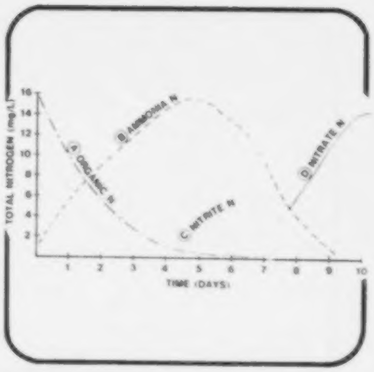
SLIDE 6


% TRANSMITTANCE
0
50
100
ABSORBANCE
>2.0
0.3
0

SLIDE 7

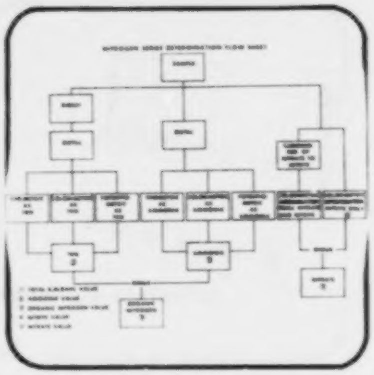

NH3 AMMONIA NH4+
BACTERIAL OXIDATION
BACTERIAL REDUCTION
NITRITE NO2-
CHEMICALS
BACTERIA
N2 ATMOSP N2
BACTERIAL REDUCTION
BACTERIAL OXIDATION
ELEC. DISCHARGE
FERTILIZERS
NITRATE NO3-
DEATH (BACTERIA)
DECOMPOSITION (BACTERIA)
HYDROLYSIS
FECAL MATTER ORG. N
URINE UREA
DEATH (BACTERIA)
FERTILIZERS
NITROGEN FIXATION (BACTERIA-ALGAE)
FERTILIZERS
ANIMAL PROTEIN ORGANIC N
FOODSTUFF
PLANT PROTEIN ORGANIC N
THE NITROGEN CYCLE

SLIDE 8



SLIDE 9

SERIES X-35, KJELDAHL NITROGEN



SLIDE 10 (LAST SLIDE)

SLIDE ______

SLIDE ______

SLIDE ______

SLIDE ______

SLIDE ______

SLIDE ______

SLIDE ______

SLIDE ______

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Total Kjeldahl Nitrogen

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 30 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control requirements.

V. ENTRY LEVEL BEHAVIOR: The participant will have completed one year of college inorganic chemistry, or equivalent.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on all the nitrogen topics (given at the end of the nitrogen topics), and will know the procedure up to the digestion step.

B. Conditions:

He/she will have heard the lecture on the Subject Matter.

C. Accepted Performance:

1. A grade of 70% on the quiz.
2. Attentiveness during the lecture, as judged by the instructor.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the Training Manual:
    a. Sources and Analysis of Organic Nitrogen
    b. Ammonia, Nitrites, and Nitrates
    c. Determination of Kjeldahl Nitrogen (Micro Apparatus-Nesslerization) (According to Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Environmental Monitoring and Support Laboratory, 1974)
2. X-35, Kjeldahl Nitrogen (10 slides); see XI, Description of Visual Materials (pages 12-3 and 12-4).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.

2. The quiz and answer sheet may be found in the IPW titled Discussion and Quiz - Nitrogen Topics. The same quiz is used for all the nitrogen topics, and is given at the end of all of the nitrogen topics.

B. Sequencing:

Classroom - discuss three outlines in Training Manual, Sources and Analysis of Organic Nitrogen; Ammonia, Nitrites and Nitrates; and Determination of Kjeldahl Nitrogen (Micro Apparatus - Nesslerization), using selected slides from X-35, Kjeldahl Nitrogen.

1. Introduction:
    - a. What the test determines
    - b. What the test does not determine
    - c. Preservation techniques
    - d. Procedure steps
        - 1) Preservation
        - 2) Digestion
        - 3) Distillation
        - 4) Determination
2. Digestion:
    - a. Macro procedure; X-35, 1
    - b. Micro procedure; X-35, 3
3. Laboratory Procedure
4. Summary

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

None

X. IPW REAGENT REQUIREMENTS:

None

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Laboratory-Kjeldahl Digestion

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 20 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant will have completed one year of college inorganic chemistry, or equivalent, and have the ability to:

A. Handle solutions of acids safely

B. Understand the terms μg, mg, g, ml, and l as metric units of mass and volume.

C. Use ordinary laboratory glassware (beakers, flasks, graduated cylinders, graduated pipets, volumetric pipets, etc.).

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on all the nitrogen topics (given at the end of the nitrogen topics), and will correctly perform the micro digestion procedure.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual and the equipment/supplies/reagents listed in sections IX and X of this IPW.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. Proper dilution of the sample, and proper addition of the digestion reagent as judged by the instructor.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

One outline in the Training Manual:

Determination of Kjeldahl Nitrogen, Micro Apparatus-Nesslerization (according to Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Environmental Monitoring and Support Laboratory, 1974)

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
2. Review the laboratory procedure by performing the student assignment.
3. The quiz and answer sheet may be found in the IPW titled Discussion and Quiz - Nitrogen Topics. The same quiz is used for all the nitrogen topics, and is given at the end of all the nitrogen topics.

B. Sequencing:

1. Classroom - inform the participants which of the standards they will prepare; i.e., for each group to do all the standards would require too much glassware. See section IX of this IPW.
2. Laboratory - have the participants do section III of the Training Manual outline, Determination of Kjeldahl Nitrogen.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: One sample. However, not every laboratory station will run the sample. Standards need not be digested.

A. For Each Station:

1 pair safety glasses (for each person at the station)

1 laboratory apron or coat (for each person at the station)

1 distilled water plastic squeeze bottle containing ammonia-free water

1 grease pencil (or other marking pencil)

paper towels or sponge

1 10 ml graduated pipet

1 pipet bulb

1 micro Kjeldahl flask containing about 10 glass beads

1 50 ml Nessler tube

B. Shared:

Digestion rack for micro Kjeldahl flask; 1 per 4 stations.

X. IPW REAGENT REQUIREMENTS:

Minimum Amounts for Each Station

(Prepare excess reagent to allow for waste, rinsing of glassware, and any desired re-runs)

Instructions for preparing the reagents may be found in section I of the Training Manual outline Determination of Kjeldahl Nitrogen (Micro Apparatus-Nesslerization)

10 ml digestion reagent

Prepare 1 liter of synthetic sample by diluting 130 ml of ammonium chloride stock solution (1.0 mg ammonia nitrogen/1.0 ml) to 1 liter; final concentration = 0.065 mg ammonia nitrogen/50 ml).

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Ammonia Nitrogen

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 20 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant will have completed one year of college inorganic chemistry, or equivalent.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on all the nitrogen topics (given at the end of the nitrogen topics), and will know the steps in the ammonia distillation procedure, the colorimetric and titrimetric determinations, and the calculations used.

B. Conditions:

He/she will have heard the lecture on the Subject Matter.

C. Accepted Performance:

1. A grade of 70% on the quiz.
2. Attentiveness during the lecture as judged by the instructor.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the Training Manual:

   Determination of Kjeldahl Nitrogen (Micro Apparatus-Nesslerization)

2. X-35, Kjeldahl Nitrogen (10 slides); see XI, Description of Visual Materials (pages 12-3 and 12-4).
3. XT-67, Determination of Ammonia Nitrogen (45 slides, 8 minute tape, script); see XI Description of Visual Materials (page 15-3, ff).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
2. The quiz and answer sheet may be found in the IPW titled Discussion and Quiz - Nitrogen Topics. The same quiz is used for all the nitrogen topics, and is given at the end of all of the nitrogen topics.

B. Sequencing:

Classroom - discuss the outline in the Training Manual, Determination of Kjeldahl Nitrogen (Micro Apparatus-Nesslerization), using selected slides from X-35, Kjeldahl Nitrogen.

1. Introduction:
    - a. Difference between total Kjeldahl nitrogen and ammonia nitrogen determinations.
    - b. Interferences
2. Distillation, macro and micro; X-35, 2, 4, 5, 6.

Classroom - discuss the determination of ammonia nitrogen using XT-67, Determination of Ammonia Nitrogen.

- a. Introduction; XT-67, 1.
- b. Topics to be covered; XT-67, 2-7.
- c. Apparatus needed to do the determination; XT-67, 8-11.
- d. Reagents needed to do the determination; XT-67, 12-21.
- e. Preparation of equipment; XT-67, 22-25.
- f. Removal of chlorine interference; XT-67, 26.
- g. Adjustment of pH sample; XT-67, 27-28.
- h. Distillation of sample; XT-67, 29-30.
- i. Titration of sample; XT-67, 31-33.
- j. Calculation of ammonia concentration using titration data; XT-67, 34-42.
- k. Summary and credits; XT-67, 43-45.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

None

X. IPW REAGENT REQUIREMENTS:

None

US EPA (LOGO)
PRESENTS

SLIDE 1

PRINTING:
DISTILLATION PROCEDURE FOR
THE ANALYSIS OF
AMMONIA NITROGEN (NH_3N)

SLIDE 2

PRINTING:
1.0 - 25 mg/l
AMMONIA NITROGEN

SLIDE 3

PRINTING:
SUMMARY OF METHOD

SLIDE 4

PRINTING:
PHOTO OF DISTILLATION APPARATUS

SLIDE 5

PRINTING:
PHOTO OF SAMPLE BEING TITRATED

SLIDE 6

PRINTING:
PHOTO OF US EPA METHODS MANUAL

SLIDE 7

PRINTING:
LABORATORY APPARATUS

SLIDE 8

PRINTING:
SAME AS 5

SLIDE 9

PHOTO OF DISTILLATION RECEIVING FLASK.

SLIDE 10

SAME AS 10

SLIDE 11

PRINTING:

REAGENTS

REAGENTS

SLIDE 12

PRINTING:

AMMONIA-FREE DISTILLED WATER

SLIDE 13

PRINTING:

AMMONIA-FREE DISTILLED WATER
BORIC ACID SOLUTION

SLIDE 14

PRINTING:

AMMONIA-FREE DISTILLED WATER
BORIC ACID SOLUTION
MIXED INDICATOR

SLIDE 15

PRINTING:

AMMONIA-FREE DISTILLED WATER
BORIC ACID SOLUTION
MIXED INDICATOR
NESSLER REAGENT

SLIDE 16

PRINTING:

AMMONIA-FREE DISTILLED WATER
BORIC ACID SOLUTION
MIXED INDICATOR
NESSLER REAGENT
BORATE BUFFER

SLIDE 17

PRINTING:

AMMONIA-FREE DISTILLED WATER
BORIC ACID SOLUTION
MIXED INIDCATOR
NESSLER REAGENT
BORATE BUFFER
SULFURIC ACID

SLIDE 18

PRINTING:

AMMONIA-FREE DISTILLED WATER

BORIC ACID SOLUTION

MIXED INDICATOR

NESSLER REAGENT

BORATE BUFFER

SULFURIC ACID

SODIUM HYDROXIDE

SLIDE 19

PRINTING:

AMMONIA-FREE DISTILLED WATER

BORIC ACID SOLUTION

MIXED INDICATOR

NESSLER REAGENT

BORATE BUFFER

SULFURIC ACID

SODIUM HYDROXIDE

SODIUM THIOSULFATE

SLIDE 20

PHOTO OF OPEN US EPA METHODS MANUAL

SLIDE 21

PRINTING:

PROCEDURE

SLIDE 22

PHOTO OF ADDITION OF WATER TO KJELDAHL FLASK

SLIDE 23

PHOTO OF ADDITION OF BOILING CHIPS TO KJELDAHL FLASK

SLIDE 24

PHOTO OF ANALYST HOLDING NESSLER'S REAGENT NEAR DISTILLATION APPARATUS

SLIDE 25

PRINTING:

CHLORINE INTERFERENCE

SLIDE 26

PHOTO OF SAMPLE pH ADJUSTMENT

SLIDE 27

PHOTO OF pH SAMPLE BEING CHECKED WITH pH PAPER

SLIDE 28

PHOTO OF SAMPLE BEING POURED INTO KJELDAHL FLASK

SLIDE 29

SAME AS 5

SLIDE 30

PHOTO OF INDICATOR BEING ADDED TO RECEIVING FLASK

SLIDE 31

SAME AS 6

SLIDE 32

SAME AS 6

SLIDE 33

PRINTING:

CALCULATION OF AMMONIA NITROGEN

SLIDE 34

PRINTING:

$$NH_3N \quad mg/l = \frac{A \times 0.28 \times 1000}{S}$$

SLIDE 35

PRINTING:

SAMPLE CALCULATION

SLIDE 36

PRINTING:

NH_3N

$mg/l = \frac{A \times 0.28 \times 1000}{400}$

SLIDE 37

PRINTING:

NH_3N

$mg/l = \frac{17.0 \times 0.28 \times 1000}{400}$

SLIDE 38

PRINTING:

NH_3N

$mg/l = \frac{17.0 \times 280}{400}$

SLIDE 39

PRINTING:

NH_3N

$mg/l = \frac{4760}{400}$

SLIDE 40

PRINTING:

NH_3N

$mg/l = 11.9$

SLIDE 41

PRINTING:

SAMPLE SIZE IMPORTANT

SLIDE 42

PRINTING:

DETERMINATION OF AMMONIA NITROGEN
BY DISTILLATION AND TITRATION

SLIDE 43

PRINTING:

AUTHOR: P. F. HALLBACH
NARRATION: C. E. SPONAGLE
TECHNICAL REVIEW: F. LUDZACK
R. KRONER

SLIDE 44

PRINTING:

CLEAN
WATER

SLIDE 45 (LAST SLIDE)

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Use of a Spectronic 20 and Preparation of Calibration Graphs

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 45 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant must use a Spectronic 20 (or equivalent) spectrophotometer, and prepare calibration graphs as part of the analyses for ammonia nitrogen, surfactants, and phenol.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of high school chemistry or equivalent, and have the ability to:

A. Perform simple calculations (addition, subtraction, multiplication, division) involving decimals.

B. Understand the terms g, mg, μg, l, and ml as being metric units of mass and volume.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will know how to zero a Spectronic 20 (or equivalent) spectrophotometer at a pre-set wavelength, and will prepare calibration graphs, using absorbance and concentration values for standards, and absorbance values for samples, obtained from the ammonia nitrogen surfactants, and phenol analyses.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual, graph paper, ruler, pen or pencil, a Spectronic 20 (or equivalent) spectrophotometer, and other equipment/supplies/reagents listed in the respective IPW's.

C. Accepted Performance:

The instructor will judge whether or not the calibration graph has been prepared properly (e.g., use of the entire x and y axes and line of best fit), whether or not the spectrophotometer has been used properly (e.g., no spilling of reagents on the instrument, and no rough handling of the instrument).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the Training Manual: Use of a Spectrophotometer

2. X-8, Use of a Spectrophotometer (16 slides); see XI, Description of Visual Materials (pages 16-4 and 16-5).

3. X-9, Calibration Graphs (6 slides); See XI, Description of Visual Materials (page 16-6).

4. XT-51, Use of the Spectronic 20 Spectrophotometer (35 slides, 10 minute tape, script); See XI, Description of Visual Materials (page 16-7, ff).

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

Review the Available Media and prepare lesson, using the Sequencing below or your own organization of the material.

B. Sequencing:

1. Classroom - discuss colorimetry, Lambert-Beer Law, and preparation of standards using X-8, Use of a Spectrophotometer.
    - a. General principles of absorbance and colorimetry; X-8, 1.
    - b. Definition of wavelength; X-8, 2.
    - c. Characteristics of wavelength; X-8, 3.
    - d. Relation of wavelength values to inches; X-8, 4.
    - e. Lambert - Beer Law; X-8, 5.
    - f. Shapes of absorption cells; X-8, 6.
    - g. Use of the EPA phosphorus procedure to exemplify the preparation of standards; X-8, 7.
    - h. Preparation of the stock phosphorus solution (known composition and concentration); X-8, 8,9, and 10.
    - i. Preparation of the standard phosphorus solution (known composition and concentration); X-8, 11, 12, and 13.
    - j. Preparation of a series of phosphorus solutions of known composition and concentration; X-8, 14, 15, and 16.
2. Classroom - discuss preparation of calibration graphs using X-9, Calibration Graphs.
    - a. Information needed to prepare a calibration graph; X-9, 1.
    - b. Drawing and labeling the axes of a calibration graph; X-9, 2. (A = absorbance, C = concentration)
    - c. Locating one point using absorbance and concentration values. (mg P/liter is an example label for the abscissa); X-9, 3.
    - d. Locating five points; X-9, 4.
    - e. Connecting the five points by the line of best fit; X-9, 5.
    - f. Determining the concentration of the sample using the calibration graph; X-9, 6.

3. Classroom - discuss sample dilution using Section V of the Training Manual outline, Use of a Spectrophotometer.

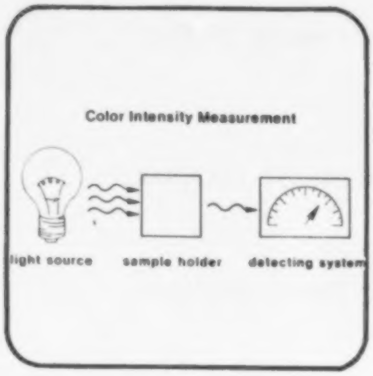
4. Classroom - discuss the use of a spectrophotometer using XT-51, Use of the Spectronic 20 Spectrophotometer.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

As specified for the ammonia nitrogen, surfactants, and phenol analyses.

X. IPW REAGENT REQUIREMENTS:

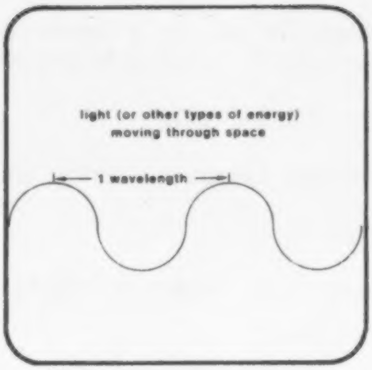
As specified for the ammonia nitrogen, surfactants, and phenol analyses.



Color Intensity Measurement

light source
sample holder
detecting system

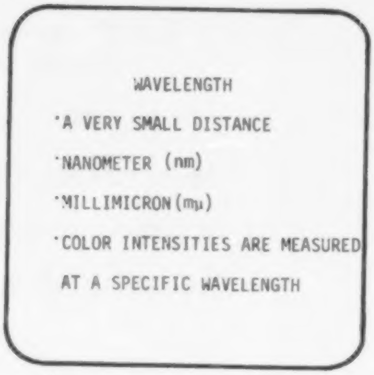
SLIDE 1



light (or other types of energy) moving through space

1 wavelength

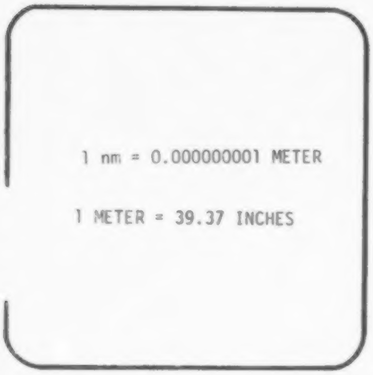
SLIDE 2



WAVELENGTH

- A VERY SMALL DISTANCE
- NANOMETER (nm)
- MILLIMICRON (mμ)
- COLOR INTENSITIES ARE MEASURED AT A SPECIFIC WAVELENGTH

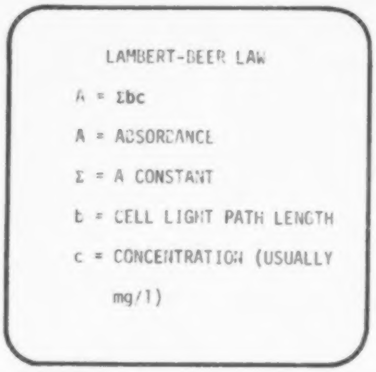
SLIDE 3



1 nm = 0.000000001 METER

1 METER = 39.37 INCHES

SLIDE 4



LAMBERT-BEER LAW

$A = \Sigma bc$

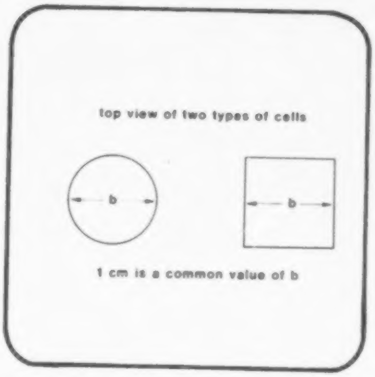
A = ABSORBANCE

Σ = A CONSTANT

b = CELL LIGHT PATH LENGTH

c = CONCENTRATION (USUALLY mg/l)

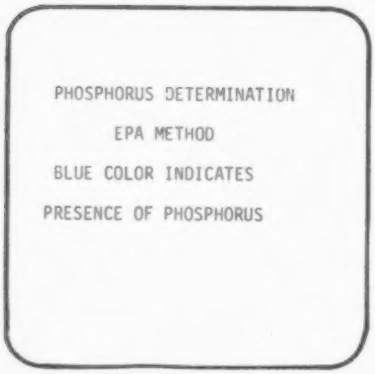
SLIDE 5



top view of two types of cells

1 cm is a common value of b

SLIDE 6



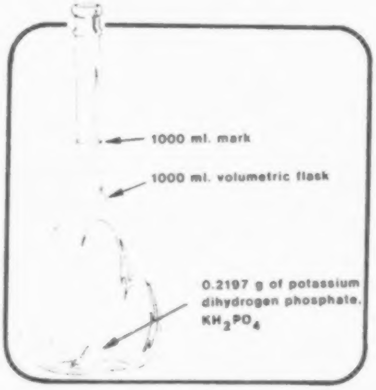
PHOSPHORUS DETERMINATION

EPA METHOD

BLUE COLOR INDICATES

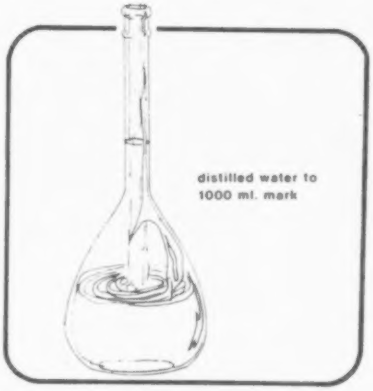
PRESENCE OF PHOSPHORUS

SLIDE 7



1000 ml. mark
1000 ml. volumetric flask
0.2197 g of potassium dihydrogen phosphate, KH2PO4

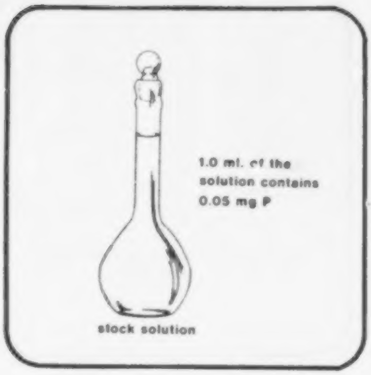
SLIDE 8



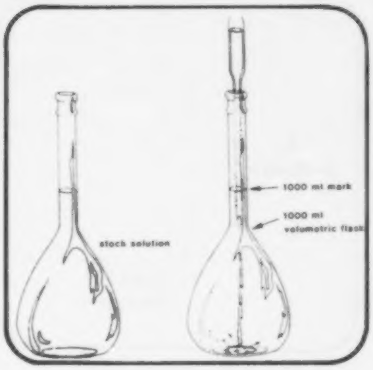
distilled water to 1000 ml. mark

SLIDE 9

SERIES X-8: USE OF A SPECTROPHOTOMETER


1.0 ml. of the
solution contains
0.05 mg P
stock solution

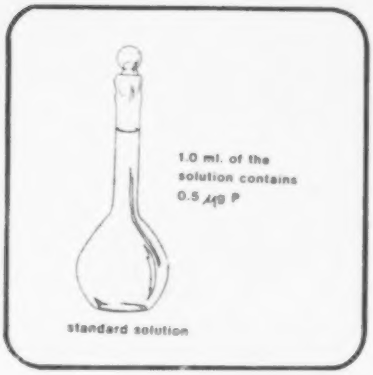
SLIDE 10


stock solution
1000 ml mark
1000 ml
volumetric flask

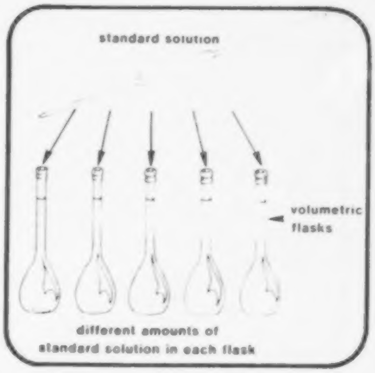
SLIDE 11

SAME AS SLIDE #9

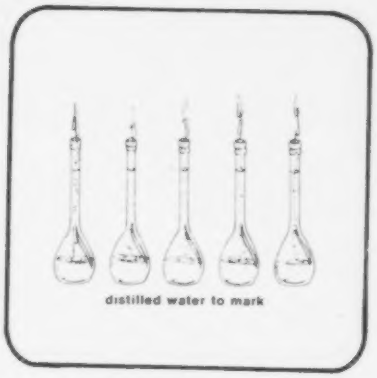
SLIDE 12


1.0 ml. of the
solution contains
0.5 μg P
standard solution

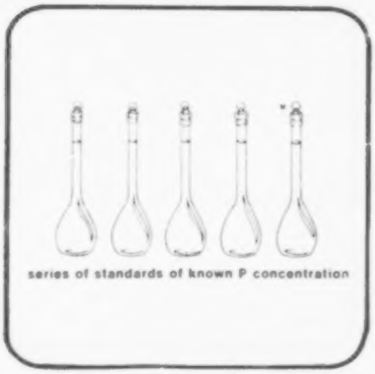
SLIDE 13


standard solution
volumetric
flasks
different amounts of
standard solution in each flask

SLIDE 14


distilled water to mark

SLIDE 15


series of standards of known P concentration

SLIDE 16 (LAST SLIDE)

SLIDE

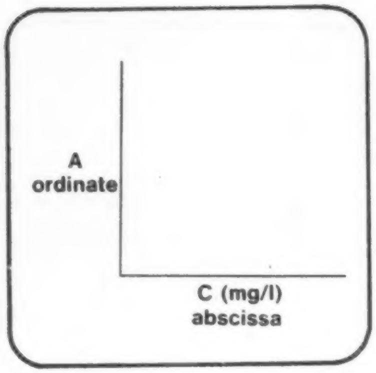
SLIDE

SERIES X-9: CALIBRATION GRAPHS

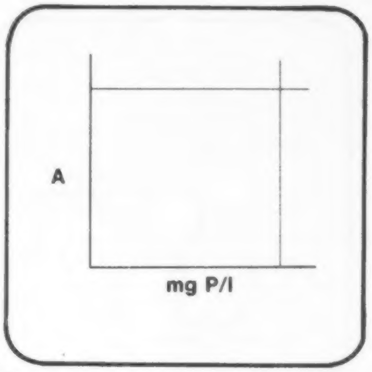
Absorbance and concentration values for each standard

Absorbance value of sample

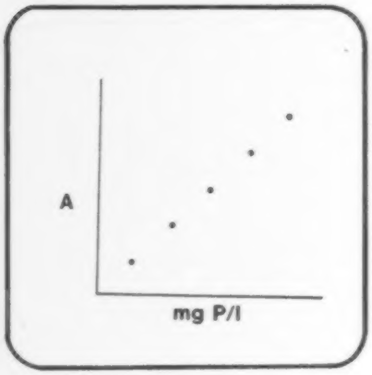
SLIDE 1


A
ordinate
C (mg/l)
abscissa

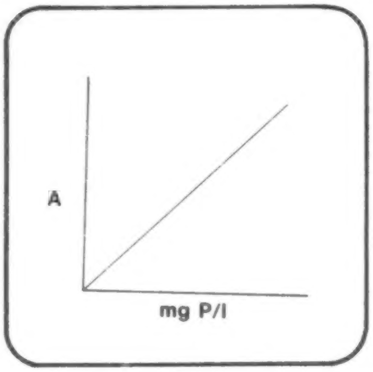
SLIDE 2



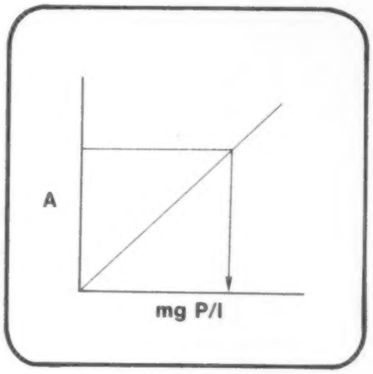
SLIDE 3



SLIDE 4



SLIDE 5



SLIDE 6 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

SERIES XT-51: USE OF A SPECTROPHOTOMETER

EPA Symbol

SLIDE 1

Environmental Protection
Agency
Office of Water Programs
National Training Center

SLIDE 2

Spectrophotometers

SLIDE 3

Spectronic 20

SLIDE 4

- % transmittance
- Absorbance
  (optical density)

SLIDE 5

- % transmittance
- absorbance

↓

concentration

SLIDE 6

Photo
of
Spectronic 20
with
Numbers on the Controls

SLIDE 7

Photo
Featuring
# 1 - On/Off Knob

SLIDE 8

Photo
Featuring
2 Cell Compartment
(closed)

SLIDE 9

SERIES XT-51: USE OF A SPECTROPHOTOMETER

Photo
Featuring
# 2 - Cell Compartment
(open)

SLIDE 10

Photo
Featuring
# 3 - Read-out Scale

SLIDE 11

Photo
Featuring
# 4 - On/Off Indicator Light

SLIDE 12

Photo
Featuring
# 5 - Wavelength Scale

SLIDE 13

Photo
Featuring
# 6 - Wavelength Selection
Knob

SLIDE 14

Photo
Featuring
# 7 - Light Control
Knob

SLIDE 15

Photo
of
Rotating Wavelength
Knob

SLIDE 16

Photo
of
Turning On/Off Knob
to "On"

SLIDE 17

Photo
of
Clock to Time Warm-up

SLIDE 18

SERIES XT-51: USE OF A SPECTROPHOTOMETER

Photo
of
Adjusting to Infinite
Absorbance

SLIDE 19

Photo
of
Rinsing Sample Cell

SLIDE 20

Photo
of
Filling Sample Cell

SLIDE 21

Photo
of
Wiping Sample Cell

SLIDE 22

Photo
of
Placing Cell in
Spectrophotometer

SLIDE 23

Photo
of
Adjusting to Zero
Absorbance

SLIDE 24

Photo
of
Recording Data

SLIDE 25

Cartoon
of
Lab with Many
Samples to be Run

SLIDE 26

- Remove cell
- Rinse with distilled water
- Rinse with solution
- Fill with solution
- Dry outside of cell
- Put cell into instrument
- Record reading

SLIDE 27

340 - 625 nm

950 nm

SLIDE 28

Photo
of
Turning On/Off Knob
to "Off"

SLIDE 29

Photo
of
Opening Access Cover
Underneath Instrument

SLIDE 30

Photo
of
Removing Phototube

SLIDE 31

Photo
of
Inserting Red Filter

SLIDE 32

Spectronic 20

SLIDE 33

Credits

SLIDE 34

EPA SYMBOL

SLIDE 35 (LAST SLIDE)

SLIDE ______

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Laboratory Briefing - Kjeldahl Distillation and Ammonia Calibration Curve

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 30 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant must have the briefing in order to be able to do the Kjeldahl distillation and prepare the calibration graph.

V. ENTRY LEVEL BEHAVIOR: The participant will have completed one year of college inorganic chemistry, or equivalent.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will know what standards he/she is to run, and whether or not he/she is to run the sample.

B. Conditions:

He/she will have heard the verbal instructions.

C. Accepted Performance:

He/she will obtain an analytical result for the assigned standards and/or sample.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

None

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Prepare the lesson, using the remaining steps in VIII, or your own organization of the material.
2. Each laboratory station will run the 0.0 standard. In addition, each station will run one other standard and the sample. Determine which stations will run the sample or standard, and which standard.
3. At least two stations should run the sample and each standard. a "composite" calibration graph will be prepared.

4. Prepare a chart on a chalkboard for the data.

B. Sequencing:

1. Classroom - make the assignments determined in VIII A. 1. above.

2. Classroom - since the supply of laboratory glassware is limited, tell the participants which stations should do the steam distillation first, and which should prepare the calibration graph first.

3. Classroom - tell the participants to post absorbancy and concentration data for the standards as soon as they are obtained.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

None

X. IPW REAGENT REQUIREMENTS:

None

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Laboratory - Kjeldahl Distillation and Ammonia Calibration Curve

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 1 hour, 10 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant will have completed one year of college inorganic chemistry, or equivalent, and have the ability to:

A. Handle solutions of acids and bases safely.

B. Understand the terms µg, mg, g, ml, and l as metric units of mass and volume.

C. Use ordinary laboratory glassware (beakers, flasks, graduated cylinders, graduated pipets, volumetric pipets, etc.).

D. Use a Spectronic 20 (or equivalent) spectrophotometer.

E. Perform simple calculations (addition, subtraction, multiplication, and division) involving decimals.

F. Prepare a calibration graph and use it to determine the concentration of a sample constituent.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on all the nitrogen topics (given at the end of the nitrogen topics), will obtain absorbance values for the assigned ammonia standards or sample, use the corresponding concentration values of standards to prepare a calibration graph, and then use the graph to determine the total Kjeldahl nitrogen ammonia concentration of the sample.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, will have heard the Laboratory Briefing, and will have the use of the Training Manual and equipment/supplies/reagents listed in sections IX and X of this IPW.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. The instructor will judge whether or not the calibration graph has been prepared properly (e.g., use of the entire x and y axes, and line of best fit).

3. Value for total Kjeldahl nitrogen ammonia concentration within ± 10% of the class average.

4. The instructor will judge whether or not the spectronic 20 (or equivalent) spectrophotometer has been used properly (e.g., no spilling of reagents on the instrument, and no rough handling of the instrument).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

One outline in the Training Manual:

Determination of Kjeldahl Nitrogen (Micro Apparatus-Nesslerization) (according to Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Environmental Monitoring and Support Laboratory, 1974).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Other items relevant to the preparation for instruction were carried out in section VIII A of the Laboratory Briefing IPW.

2. Prepare the equipment/supplies/reagents listed in sections IX and X of this IPW.

3. Since the procedure is highly sensitive to contamination, it should be carried through by a staff member prior to the start of the course.

4. Rinse the steam distillation apparatus and Kjeldahl flasks with Nessler reagent and then distilled water.

5. Just prior to the laboratory session, a staff member should bring the water in the steam distillation apparatus to a boil. (See section IV B. 1. in the Training Manual outline).

6. The quiz and answer sheet may be found in the IPW titled Discussion and Quiz - Nitrogen Topics. The same quiz is used for all the nitrogen topics, and is given at the end of all the nitrogen topics.

B. Sequencing:

1. Laboratory - remind the participants who is to do the steam distillation first, and who is to prepare the calibration graph first.

2. Laboratory - tell the students to bring the samples for steam distillation to the apparatus, and that the instructor will pour the samples into the apparatus.

   Note: The order of doing steps 3 and 4 has already been mentioned.

3. Laboratory - have the participants do section IV of the Training Manual outline. (The instructor will do step IV A. 2.)

4. Laboratory - have the participants do section V of the Training Manual outline.

5. Classroom - have the participants do sections VI and VII of the Training Manual outline.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: The 0.0 standard and the sample or other standard.

A. For Each Station:

1 pair safety glasses (for each person at the station)

1 laboratory apron or coat (for each person at the station)

1 distilled water plastic squeeze bottle containing ammonia-free water

1 grease pencil (or other marking pencil)

paper towels or sponge

tissues (for wiping the spectrophotometer cell)

1 50 ml Nessler tube; 1 for each sample/standard

1 clean rubber stopper; 1 for each Nessler tube to allow mixing. (Unless grossly contaminated, stoppers may be cleaned by boiling in water and rinsing with distilled water.)

1 rack for holding the Nessler tubes; a beaker may be used

2 10 ml graduated pipets

1 1 ml volumetric pipet

1 pipet bulb

1 50 ml graduated cylinder

1 1 cm cell for the Spectronic 20 (or equivalent) spectrophotometer

B. Shared:

Micro Kjeldahl steam distillation apparatus; 1 per 6 stations. Also, 1 pair asbestos gloves and matches.

Spectronic 20 (or equivalent) spectrophotometer; 1 per 4 stations, and a large beaker for rinsing the cells.

X. IPW REAGENT REQUIREMENTS:

Minimum Amounts for Each Station
(Prepare excess reagent to allow for waste, rinsing of glassware, and any desired re-runs)

Instructions for preparing the reagents may be found in section I of the Training Manual outline Determination of Kjeldahl Nitrogen (Micro Apparatus-Nesslerization)

10 ml sodium hydroxide-sodium thiosulfate (at the distillation apparatus)

5 ml boric acid (at the distillation apparatus)

1 ml Nessler reagent; 1 ml per group per sample/standard

Ammonium chloride standard solution; volumes as assigned.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Discussion and Quiz - Nitrogen Topics

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 20 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: This discussion and quiz summarizes the previous lecture and laboratory sessions.

V. ENTRY LEVEL BEHAVIOR: The participant will have successfully met the Accepted Performance listed in all other IPW's dealing with the nitrogen topics.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take part in a discussion on all previous nitrogen topics, and will take the quiz referred to in all the IPW's on the nitrogen topics.

B. Conditions:

He/she will have successfully met the Accepted Performances listed in all other IPW's dealing with the nitrogen topics.

C. Accepted Performance:

1. Attentiveness during the discussion as judged by the instructor.

2. A grade of 70% on the quiz.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

Items cited in the other IPW's on the nitrogen topics.

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media cited in the other nitrogen topics IPW's and prepare the discussion.

2. Answering questions relative to the various laboratory procedures is the main purpose of the discussion.

3. Reproduce the attached quiz; one copy for each participant, plus any desired file copies. See pages 19-3 and 19-4.

B. Sequencing:

Classroom - discuss potential problems in the determination.

1. Ammonia must be absent from the:
   a. Distilled water
   b. Steam distillation apparatus
   c. Kjeldahl flasks
   d. Reagents
2. Use of blanks:
   a. Not steam distilled, not digested
   b. Steam distilled, digested

Classroom - give the quiz and then read the correct answers from the attached answer sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

1 copy of the quiz for each participant

X. IPW REAGENT REQUIREMENTS:

None

NAME____________________

QUIZ

Nitrogen Cycle, Kjeldahl Nitrogen, Ammonia Nitrogen

Check the correct answer or fill in the missing word(s) as indicated.

1. Three sources of organic nitrogen (either of natural or industrial origin) are ____________________, ____________________, and ____________________.
2. In the Kjeldahl determination, if the nitrogen concentration is below 1.0 mg/l, it is determined by ____________________. If it is above 1.0 mg/l, it is determined by ____________________.
3. In the Kjeldahl determination, the digestion mixture consists of ____________________ ____________________ and ____________________.
4. During the Kjeldahl digestion, all nitrogen in the sample is converted to ____________________.
5. In the Kjeldahl determination, Nesslerization produces a ____________________ color in samples having a small amount of nitrogen.
6. In the Kjeldahl determination, distilled water used to dilute the sample/standards must be ____________________ free.

NAME ______________________

QUIZ - ANSWER SHEET

Nitrogen Cycle, Kjeldahl Nitrogen, Ammonia Nitrogen

Check the correct answer or fill in the missing word(s) as indicated.

1. Three sources of organic nitrogen (either of natural or industrial origin) are dead animal & plant residue, animal wastes, and food processing; wastewater; or (1)

2. In the Kjeldahl determination, if the nitrogen concentration is below 1.0 mg/l, it is determined by colorimetry. If it is above 1.0 mg/l, it is determined by titration.

3. In the Kjeldahl determination, the digestion mixture consists of potassium sulfate concentrated sulfuric acid and mercuric sulfate.

4. During the Kjeldahl digestion, all nitrogen in the sample is converted to ammonia.

5. In the Kjeldahl determination, Nesslerization produces a pale yellow or yellow color in samples having a small amount of nitrogen.

6. In the Kjeldahl determination, distilled water used to dilute the sample/ standards must be ammonia free.

(1) autotrophic organisms, heterotrophic organisms, pharmaceutical wastes, plastics, chemical intermediates or products, dyes.

Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For "fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Chemical Oxygen Demand

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 2 hours, 50 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of college analytical chemistry or equivalent, and have the ability to:

A. Use ordinary laboratory glassware (beakers, flasks, graduated cylinders, graduated pipets, volumetric pipets, etc.).

B. Understand the terms μg, mg, g, ml, and l as metric terms of mass and volume.

C. Perform simple calculations (addition, subtraction, multiplication, division) involving decimals.

D. Perform a titration.

E. Handle solutions of acids safely.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on the Subject Matter, will standardize ferrous ammonium sulfate (FAS) solution, and will determine the chemical oxygen demand (COD) of an influent, effluent, or blank.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual, and the equipment/supplies/reagents listed in sections IX and X of this IPW.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. Ml of FAS used in the standardization within ± 0.3 ml of a majority of the class.

3. COD value within ± 20 mg/l of the class average for the sample.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in the Training Manual:
    a. Chemical Oxygen Demand and COD/BOD Relationships
    b. Laboratory Procedure for Routine Level Chemical Oxygen Demand (according to Standard Methods for the Examination of Water and Wastewater, 14th edition, page 550, 1975)
2. XT-27, Chemical Oxygen Demand (60 slides, 17 minute tape, script); see XI, Description of Visual Materials (page 20-10, ff).
3. Information relevant to mercury disposal.
    a. Dean, et al., Disposal of Mercury Wastes from Water Laboratories, Environmental Science and Technology, volume 5, no. 10, 1971.
    b. Maag and Hecker, Recovery of Mercury in Solution, Journal Environmental Quality, volume 1, no. 2, 1972.
4. Information relevant to saline COD samples.

    Burnes and Marshall, Correction for Chloride Interference in the Chemical Oxygen Demand Test, Journal WPCF, volume 37, no. 12, 1965.

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
2. Review the laboratory procedure by performing the student assignment.
3. Reproduce the attached quiz; one copy for each participant, plus any desired file copies. See pages 20-6 through 20-9.
4. Prepare the equipment/supplies/reagents listed in Sections IX and X of this IPW.
5. Prepare reflux apparatus for use. See Section II in the outline, Laboratory Procedure for Rountine Level Chemical Oxygen Demand.
6. Determine which stations will run the influent, effluent, or blank. The following assignments might be used for an unknown effluent and influent sample:

    ¼ of the stations - will do a 100% effluent sample (i.e., 20 ml of sample)

    ¼ of the stations - will do a 50% dilution of the influent sample (i.e., 10 ml of sample + 10 ml of distilled water)

    ¼ of the stations - will do a 25 % dilution of the influent (i.e., 5 ml of sample + 15 ml of distilled water)

    ¼ of the stations - will do a blank (i.e., 20 ml of distilled water)

B. Sequencing:

1. Classroom - discuss COD using the outline Chemical Oxygen Demand and COD/BOD Relationships.
    a. Definition
    b. Contrast to BOD
    c. Correlation of COD/BOD
2. Classroom - discuss COD using XT-27, Chemical Oxygen Demand
    a. Introduction; XT-27, 1-3.
    b. Topics to be covered; XT-27, 4.
    c. Discussion of the determination, and information obtained from it; XT-27, 5-12.
    d. Potassium dichromate as the oxidizing agent in the test; XT-27, 13-18.
    e. Discussion of the blank; XT-27, 19-20.
    f. Oxidizing conditions; XT-27, 21-22.
    g. Potassium dichromate concentration in the determination; XT-27, 23-24.
    h. Chemical reaction in the oxidation step XT-27, 25.
    i. Oxidizing conditions; XT-27, 26-28.
    j. Titration with ferrous ammonium sulfate; XT-27, 29-33.
    k. Summary of the determination; XT-27, 34-35.
    l. Calculations, XT-27, 36-42.
    m. Limitations and interferences; XT-27, 43-51.
    n. Sample treatment; XT-27, 52-53.
    o. Usefulness of data obtained from the determination; XT-27, 54-57.
    p. Summary and credits; XT-27, 58-60.
3. Classroom - discuss the EPA method for determining COD using the outline, Chemical Oxygen Demand and COD/BOD Relationships.
4. Classroom - discuss disposal of test materials using the items in VII A. 3.
5. Laboratory - make the assignments determined in VIII A. 5. above.

Caution the participants about the use of sulfuric acid. Tell the the participants that section II of the outline Laboratory Procedure for Routine Level Chemical Oxygen Demand has already been done.

6. Laboratory - have the participants do sections IV A through IV J of the of the same outline. (The instructor will turn off the heat after a 2 hour reflux period.)

7. Laboratory - the next day (as indicated on the agenda), have the participants do sections IV L through IV Q of the same outline.

8. Laboratory - have the participants do section III of the same outline.

9. Classroom - have the participants who ran the blank, report A and AN (see formula in section V of the same outline).

10. Classroom - have the participants calculate BN for their sample (see same formula).

11. Classroom - have the participants calculate the COD for their sample.

12. Classroom - final discussion and questions.

13. Classroom - give the quiz and then read the correct answers from the attached answer sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: One influent or effluent sample, or blank.

A. For Each Station:

1 pair safety glasses (for each person at the station)

1 laboratory apron or coat (for each person at the station)

1 distilled water plastic squeeze bottle

1 grease pencil (or other marking pencil)

paper towels or sponge

1 50 ml buret

1 small funnel (to fit into the buret)

1 buret clamp

1 ring stand

2 150-250 ml beakers

1 100 ml graduated cylinder

2 50 ml graduated cylinders

1 reflux apparatus, consisting of:

  1 250-500 ml Erlenmeyer flask, 24/40 ground glass neck, containing 6-10 glass beads (or equivalent)

  1 Liebig condenser (or equivalent), 300 mm jacket, 24/40 ground glass end and tubing

  1 hot plate

1 250-500 ml wide mouth Erlenmeyer flask (or sample volumetric assigned)

1 20 ml volumetric pipet

1 10 ml graduated pipet

1 10 ml volumetric pipet

1 pipet bulb

B. Shared:

1. Pan of ice water (to cool reaction flask), 1 per 2 stations
2. Scoop (to measure 0.4g mecuric sulfate), 1 per 2 stations

X. IPW REAGENT REQUIREMENTS:

Minimum Amounts for Each Station
(Prepare excess reagent to allow for waste, rinsing of glassware, and any desired re-runs).

Instructions for preparing the reagents may be found in section I of the Training Manual outline, Laboratory Procedure for Routine Level Chemical Oxygen Demand.

0.4 g mecuric sulfate

33 ml concentrated sulfuric acid

20 ml 0.250 N potassium dichromate

27 ml sulfuric acid-silver sulfate

4-6 drops ferroin indicator

50 ml 0.1 N ferrous ammonium sulfate

20 ml of influent or effluent (depending on the assignment)

NAME________________________

QUIZ
Chemical Oxygen Demand

Check the correct answer(s) or fill in the missing word(s) as indicated.

1. In the COD test, the oxidizing system includes:

   _____hypochlorous acid and oxygen

   _____enzymes and oxygen

   _____50% sulfuric acid, potassium dichromate, and possibly a catalyst

   _____phosphoric acid and iodic acid

   _____oxygen and catalyst

2. The COD test measures:

   _____the oxygen equivalent/demand of organic matter

   _____buffer capacity

   _____the amount of inorganic material present in the sample

   _____the ultimate BOD

   _____dissolved and suspended solids

3. All organic compounds:

   _____are completely oxidized in the COD test

   _____require the use of a catalyst in the COD test

   _____do not give the same degree of analytical response in the COD test

   _____are completely reduced in the COD test

   _____have equal values for COD and BOD

4. In the COD titration, the titration color change is:

   _____blue to colorless

   _____colorless to pink

   _____brown to green

   _____blue-green to red-brown

   _____orange to red

5. The COD sample is titrated with:

   _____potassium dichromate

NAME________________________

_____50% sulfuric acid

_____silver sulfate solution

_____o-phenanthroline

_____ferrous ammonium sulfate

6. In the COD test, the sample is refluxed for 2 hours.

_____T

_____F

7. One advantage of the COD test is that it gives the same numerical result as the BOD test, but in a much shorter time.

_____T

_____F

8. ______________________ ______________________ is added to the COD sample to take care of chloride interference.

9. ______________________ ______________________ is used as a catalyst in the COD test.

10. The ferrous ammonium sulfate used in the COD test is stable for long periods of time.

_____T

_____F

NAME______________________

QUIZ - ANSWER SHEET
Chemical Oxygen Demand

Check the correct answer(s) or fill in the missing word(s) as indicated.

1. In the COD test, the oxidizing system includes:

_____hypochlorous acid and oxygen

_____enzymes and oxygen

__X__50% sulfuric acid, potassium dichromate, and possibly a catalyst

_____phosphoric acid and iodic acid

_____oxygen and catalyst

2. The COD test measures:

__X__the oxygen equivalent/demand of organic matter

_____buffer capacity

_____the amount of inorganic material present in the sample

_____the ultimate BOD

_____dissolved and suspended solids

3. All organic compounds:

_____are completely oxidized in the COD test

_____require the use of a catalyst in the COD test

__X__do not give the same degree of analytical response in the COD test

_____are completely reduced in the COD test

_____have equal values for COD and BOD

4. In the COD titration, the titration color change is:

_____blue to colorless

_____colorless to pink

_____brown to green

__X__blue-green to red-brown

_____orange to red

5. The COD sample is titrated with:

_____potassium dichromate

NAME______________________

_____50% sulfuric acid

_____silver sulfate solution

_____o-phenanthroline

__X__ferrous ammonium sulfate

6. In the COD test, the sample is refluxed for 2 hours.

__X__T

_____F

7. One advantage of the COD test is that it gives the same numerical result as the BOD test, but in a much shorter time.

_____T

__X__F

8. __MECURIC__ __SULFATE__ is added to the COD sample to take care of chloride interference.

9. __SILVER__ __SULFATE__ is used as a catalyst in the COD test.

10. The ferrous ammonium sulfate used in the COD test is stable for long periods of time.

_____T

__X__F

Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For "fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

XI. DESCRIPTION OF VISUAL MATERIALS

SERIES XT-27, CHEMICAL OXYGEN DEMAND

EPA LOGO PRESENTS

SLIDE 1

PRINTING:

CHEMICAL DETERMINATION
OF ORGANIC
OXYGEN DEMAND

US EPA

SLIDE 2

PRINTING:

→COD←

CHEMICAL
OXYGEN
DEMAND
TEST

SLIDE 3

PHOTO OF INDUSTRY AND RECEIVING STREAM.

SLIDE 4

PRINTING:

ORGANIC COMPOUNDS → OXIDIZED → CARBON DIOXIDE
WATER

SLIDE 5

PRINTING:

ORGANIC SUBSTANCES
BIO-DEGRADABLE
NON BIO-DEGRADABLE

SLIDE 6

PRINTING:

BIOLOGICALLY
OXIDIZABLE

RELATIVELY
BIOCHEMICALLY
INERT

SLIDE 7

PRINTING:

GLUCOSE BIO-DEGRADABLE
LIGNIN NON BIO-DEGRADABLE

LIGNIN → OXIDIZED → COMPLETELY

SLIDE 8

PRINTING:

PLANT INFLUENT SAMPLE

CHEMICAL OXYGEN DEMAND = 430 mg/l
BIOCHEMICAL OXYGEN DEMAND = 220 mg/l

SLIDE 9

SERIES XT-27, CHEMICAL OXYGEN DEMAND

PHOTO OF POND

SLIDE 10

PRINTING:

COD TEST	3 HOURS
BOD TEST	5 DAYS

SLIDE 11

COD/BOD

RELATIONSHIPS & CORRELATION FACTORS

SLIDE 12

PRINTING:

HISTORY OF THE COD TEST

SLIDE 13

PRINTING:

POTASSIUM PERMANGANATE

CERIC SULFATE

POTASSIUM IODATE

POTASSIUM DICHROMATE

SLIDE 14

PHOTO OF BOTTLE OF POTASSIUM DICHROMATE AND WEIGHING BOAT CONTAINING DICHROMATE.

SLIDE 15

PHOTO OF ANALYST PERFORMING WEIGHING.

SLIDE 16

PHOTO OF DICHROMATE SOLUTION BEING PREPARED IN A VOLUMETRIC FLASK.

SLIDE 17

PRINTING:

ORGANIC MATTER + DICHROMATE +
CHO $Cr_2O_7^=$
SULFURIC ACID $\xrightarrow{\hspace{2em}}$
H^+
REDUCED CHROMIUM ION + CARBON DIOXIDE & WATER
Cr^{+3}

SLIDE 18

PRINTING:

OXYGEN REQUIREMENTS BY

OXIDATION OF ORGANIC MATTER

SLIDE 19

PHOTO OF SAMPLE AND
BLANK IN FLASK.

SLIDE 20

PRINTING:

STRONGLY ACID
CONCENTRATED SULFURIC

ELEVATED TEMPERATURE
BOIL FOR 2 HOURS

SLIDE 21

PHOTO OF COD SAMPLES AND
BLANK BEING DIGESTED.

SLIDE 22

PRINTING:

NORMALITY OR CONCENTRATION OF
DICHROMATE 0.25N.

SLIDE 23

PRINTING:

DICHROMATE SOLUTION
1 ML = 2 MG OXYGEN

SLIDE 24

PRINTING:

ORANGE
DICHROMATE + ORGANIC MATTER +
$(+6\ -2)^{-2}$ CHO
(Cr_2O_7)

SULFURIC ACID $\longrightarrow$
H^{+1}

GREEN
RELEASED CHROMIUM ION +
Cr^{+3} CARBON DIOXIDE & WATER

SLIDE 25

PHOTO OF POTASSIUM DICHROMATE
BEING PIPETTED INTO SAMPLE.

SLIDE 26

PHOTO OF DIGESTION IN PROGRESS.

SLIDE 27

SERIES XT-27, CHEMICAL OXYGEN DEMAND

PRINTING:

ORANGE
DICHROMATE + ORGANIC MATTER +
$(+6\ -2)^{-2}$ CHO
Cr_2O_7

SULFURIC ACID $\longrightarrow$
H^{+1}

GREEN
RELEASED CHROMIUM + CARBON
Cr^{+3} DIOXIDE & WATER

SLIDE 28

PHOTO OF SAMPLE READY FOR TITRATION.

SLIDE 29

PRINTING:

$6\ Fe^{+2} + (\overset{+6\ -2}{Cr_2O_7})^{-2} + 14\ H^{+1} \longrightarrow$

IRON II REDUCES CHROMIUM IN DICHROMATE

$2Cr^{+3} + 6Fe^{+3} + 7H_2O$

SLIDE 30

PHOTO OF FLASK CONTAINING POTASSIUM DICHROMATE AND ONE CONTAINING FERROUS AMMONIUM SULFATE.

SLIDE 31

PHOTO OF INDICATOR BEING ADDED TO FLASK.

SLIDE 32

PHOTO SHOWING TITRATION END POINT.

SLIDE 33

PRINTING:

ORGANIC MATTER + DICHROMATE +
CHO Cr_2O_7

SULFURIC ACID $\longrightarrow$
H^{+}

REDUCED CHROMIUM + CARBON
ION Cr^{+3} DIOXIDE & WATER

SLIDE 34

PRINTING:

$6\ Fe^{+2} + (\overset{+6\ -2}{Cr_2O_7})^{-2} + 14\ H^{+1} \longrightarrow$

IRON II REDUCES CHROMIUM IN DICHROMATE

$2\ Cr^{+3} + 6\ Fe^{+3} + 7\ H_2O$

SLIDE 35

$$\text{COD mg/l} = \frac{(a - b)\ N \times 8000}{\text{ml of sample}}$$

SLIDE 36

$$\text{COD mg/l} = \frac{(a - b)\ N \times 8000}{\text{ml of sample}}$$

COD = CHEMICAL OXYGEN DEMAND
FROM DICHROMATE

SLIDE 37

$$\text{COD mg/l} = \frac{(a - b)\ N \times 8000}{\text{ml of sample}}$$

a = ml Fe $(NH_4)_2\ (SO_4)_2$

USED FOR BLANK

SLIDE 38

$$\text{COD mg/l} = \frac{(a - b)\ N \times 8000}{\text{ml of sample}}$$

b = ml Fe $(NH_4)_2\ (SO_4)_2$

USED FOR SAMPLE

SLIDE 39

$$\text{COD mg/l} = \frac{(a - b)\ N \times 8000}{\text{ml of sample}}$$

(a - b) = ml of Fe $(NH_4)_2\ (SO_4)_2$

TO TITRATE $K_2Cr_2O_7$

OXIDIZING THE SAMPLE

SLIDE 40

$$\text{COD mg/l} = \frac{(a - b)\ N \times 8000}{\text{ml of sample}}$$

N = NORMALITY OF Fe $(NH_4)_2\ (SO_4)_2$

SLIDE 41

PRINTING:

$$\text{COD mg/l} = \frac{(a-b)\ N \times 8000}{\text{ml of sample}}$$

8000

8 1000

OXYGEN:

AT. WGT. = 16

VALANCE = 2

EQUIVALENT WGT. = 16/2 = 8

SLIDE 42

PRINTING:

LIMITATIONS AND INTERFERENCES

SLIDE 43

PRINTING:

COD TEST

1. DOES NOT DISTINGUISH BIOLOGICALLY INERT ORGANIC MATTER.
2. DOES NOT MEASURE THE RATE OF BIOLOGICAL STABILIZATION.

SLIDE 44

PRINTING:

- STRAIGHT CHAIN ALIPHATICS
- AMMONIA
- AROMATIC HYDROCARBONS
- PYRIDINE

SLIDE 45

SERIES XT-27, CHEMICAL OXYGEN DEMAND

PRINTING:

LOW MOLECULAR WEIGHT FATTY ACIDS

↘

OXIDIZED

↘

IN PRESENCE OF SILVER ION

SLIDE 46

PRINTING:

SILVER SULFATE

Ag_2SO_4

CHLORIDES

BROMIDES

IODIDES

SLIDE 47

PRINTING:

MERCURIC SULFATE

$HgSO_4$

COMPLEXING AGENT FOR CHLORIDES AND OTHER HALOGENS

SLIDE 48

PRINTING:

$$6\ Cl^- + Cr_2O_7^{=} + 14H^+ \xrightarrow{\text{OXIDATION OF CHLORINE}} 3\ Cl_2 + 2\ Cr^{+3} + 7H_2O$$

SLIDE 49

PHOTO OF MERCURIC SULFATE BEING ADDED TO SAMPLE.

SLIDE 50

PRINTING:

- AMMONIA
- AROMATIC HYDROCARBONS
- PYRIDINE

SLIDE 51

PHOTO OF SAMPLE IN BLENDER.

SLIDE 52

PHOTO OF SULFURIC ACID BEING ADDED TO SAMPLE.

SLIDE 53

PHOTO OF WASTEWATER TREATMENT PLANT.

SLIDE 54

SERIES XT-27, CHEMICAL OXYGEN DEMAND

PRINTING:

PLANT INFLUENT SAMPLE

CHEMICAL OXYGEN DEMAND =

430 mg/liter

BIOCHEMICAL OXYGEN DEMAND =

220 mg/liter

COD/BOD = 430/220 = 1.9

SLIDE 55

PRINTING:

COD/BOD RATIOS MUNICIPAL WASTEWATERS

SAMPLE	COD_{AgHg}	BOD_5	COD/ BOD
UP-STREAM	30	3.1	9.7
PLANT INFLUENT	430	220.	1.9
ACT. ST. EFFLUENT	85	19.	4.5
1000 YDS BELOW OUTFALL	70	16.	4.4
5 MI BELOW OUTFALL	35	4.8	7.3
STAB. SURF. WATER	20	2.0	10

SLIDE 56

PRINTING:

COD/BOD RATIOS INDUSTRIAL WASTEWATERS

	COD AgHg	BOD5	COD/BOD
DISTILLERY	3100	1600	1.9
SOAP MFG.	530	300	1.8
TEXTILE DYE	38000	280	135.
OIL REFINERY	48000	8000	6.0
UREA (mg/mg calc.)	0	3.1	——

SLIDE 57

PRINTING:

BASIC PRINCIPLES OF COD TEST

APPLICATION OF TEST FOR ESTIMATION OF POLLUTIONAL STRENGTH OF MUNICIPAL AND INDUSTRIAL WASTEWATER

SLIDE 58

CREDITS

SLIDE 59

PRINTING:

CLEAN WATER

SLIDE 60 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Surfactants

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 2 hours, 55 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control requirements.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of college organic chemistry, or equivalent, and have the ability to:

A. Handle solutions of acids and bases safely.

B. Handle organic solvents safely.

C. Use a separatory funnel to perform liquid-liquid extractions.

D. Use a Spectronic 20 (or equivalent) spectrophotometer.

E. Prepare a calibration graph and use it to determine the concentration of a sample constituent.

F. Understand the terms μg, mg, g, ml, and l as metric units of mass and volume.

G. Perform simple calculations (addition, subtraction, multiplication, division) involving decimals.

H. Use ordinary laboratory glassware (beakers, flasks, graduated cylinders, graduated pipets, volumetric pipets, etc.)

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on the Subject Matter, will obtain absorbance values for the assigned surfactant standards, use them with the corresponding concentration values to prepare a calibration graph, and then use the graph to determine the surfactant concentration of the sample.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual, and the equipment/supplies/reagents listed in sections IX and X of this IPW.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. The instructor will judge whether or not the calibration graph has been prepared properly (e.g., use of the entire x and y axes,

and line of best fit).

3. Value for surfactant concentration of sample within ± 10% of class average.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in the Training Manual:
    a. Determination of surfactants
    b. Laboratory Determination of Surfactants (according to Standard Methods for the Examination of Water and Wastewater, 14th edition, page 600, 1975)
2. X-34, Surfactants (20 slides); See XI, Description of Visual Materials (pages 21-7 ff).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Order LAS Standard from USEPA, EMSL, Quality Assurance Branch, Cincinnati, Ohio 45268.
2. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
3. Review the laboratory procedure by performing the student assignment.
4. Reproduce the attached quiz; one copy for each participant, plus any desired file copies. See pages 21-5 and 21-6.
5. Prepare the equipment/supplies/reagents listed in Section IX and X of this IPW.
6. Obtain a sample of municipal wastewater treatment effluent. A maximum of 100 ml per laboratory station will be needed.
7. Each laboratory station will run the 0.0 standard, 2 other standards, and the sample. Determine which stations will run which other standards. A "composite" calibration graph will be prepared.
8. Prepare a chart on a chalkboard for the data.

B. Sequencing:

1. Classroom - discuss the determination of surfactants using X-34, Surfactants.
    a. Definitions relevant to the discussion; X-34, 1.
    b. Materials used as builders; X-34, 2.

c. Sources of surfactant wastes; X-34, 3.

d. Magnitude of surfactant usage; X-34, 4.

e. Types of surfactants; X-34, 5.

f. Equations showing the formation of anionic, cationic, and non-ionic surface active agents in water; X-34, 6, 7, and 8.

g. Hydrophobic and hydrophilic portions of a surfactant molecule; X-34, 9.

h. How water molecules form a layer which is broken up by addition of a surfactant; X-34, 10.

i. How surfactant molecules orinet themselves around grease particles, thus emulsifying them; X-34, 11.

j. Effects of surfactants on water quality. ABS is hard to degrade; LAS, easy. X-34, 12.

k. Formulas for LAS and ABS surfactants; X-34, 13 and 14.

l. Degradability of LAS and ABS with time; X-34, 15.

m. General information about the NPDES method of surfactant analysis; X-34, 16.

n. A general outline of the analytical procedure; X-34, 17.

o. Reactions in the analytical procedure; X-34, 18 and 19.

p. Interferences; X-34, 20.

2. Classroom - make the assignments determined in VIII A. 5. above.
3. Classroom - tell the participants to post absorbance and concentration data for the standards as soon as they are available.
4. Laboratory - have the participants do Section II of the Training Manual outline, Laboratory Determination of Surfactants.
5. Classroom - determine which absorbance values will be used by the class to prepare the calibration graph.
6. Classroom - have the participants do Section III of the outline.
7. Classroom - final discussion and questions.
8. Classroom - give the quiz and then read the correct answers from the attached sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 1 sample, the 0.0 standard, and 2 other standards.

A. For Each Station:

1 pair safety glasses (for each person at the station)
1 laboratory apron or coat (for each person at the station)
1 distilled water plastic squeeeze bottle
1 grease pencil (or other marking pencil)
paper towels or sponge
tissues (for wiping the spectrophotometer cell)

1 100 ml graduated cylinder

1 10 ml graduated cylinder

4 200 or 250 ml separatory funnels with Teflon stopcocks

Ring stands, rings, clamps, racks, etc., to support the separatory funnels.

Volumetric or graduated pipets to prepare the assigned standards.

1 pipet bulb

4 125 ml Erlenmeyer flasks

4 100 ml volumetric flasks

1 small filtering funnel, about 5 cm in diameter

4 pieces medium porosity filter paper (for use with the filtering funnel)

1 1 cm cell for the Spectronic 20 (or equivalent) spectrophotometer

4 small pieces of glass wool

B. Shared:

Spectronic 20 (or equivalent) spectrophotometer; 1 per 4 stations, with a large beaker for rinsing the cell.

Chalkboard, chalk, and erasers for posting data.

X. IPW REAGENT REQUIREMENTS:

Minimum Amounts for Each Station

(Prepare excess reagent to allow for waste, rinsing of glassware, and and desired re-runs).

Instructions for preparing the reagents may be found in Section I of the Training Manual outline, Laboratory Determination of Surfactants.

1 ml phenolphthlein*

5 ml 1 N sodium hydroxide*

1 ml 1 N sulfuric acid*

100 ml methylene blue

200 ml wash solution

440 ml chloroform

Standard linear alkylate sulfonate (LAS); volumes as assigned. LAS standard may be obtained by writing to: USEPA, EMSL, Quality Assurance Branch, Cincinnati, Ohio 45268.

*In eyedropper bottles

NAME ______________________________

QUIZ

Surfactants

Check the correct answer or fill in the missing word(s) as indicated.

1. Three types of surfactants are ____________, ____________, and ____________.

2. Two problems caused by surfactants in surface waters are ____________, and ____________.

3. LAS compounds are more easily biodegraded than ABS compounds.

   _______T

   _______F

4. What compound is used in the analysis of surfactants to form a blue complex with the surfactants? ____________________

5. List three interferences in the MBAS method of determining surfactants.

   __________________, ________________, __________________ NAME ______________________________

QUIZ - ANSWER SHEET
Surfactants

Check the correct answer or fill in the missing word(s) as indicated.

1. Three types of surfactants are anionic, cationic, and nonionic.

2. Two problems caused by surfactants in surface waters are foaming, and eutrophication, or persistance in biological treatment.

3. LAS compounds are more easily biodegraded than ABS compounds.

   X T

   ____ F

4. What compound is used in the analysis of surfactants to form a blue complex with the surfactants? methylene blue, or MB

5. List three interferences in the MBAS method of determining surfactants.

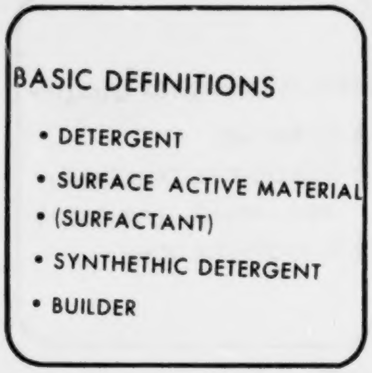
   sulfates, carboxylates, phosphates; or phenols, cyantes, chlorides, nitrates, thiocyanates.

Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For "fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

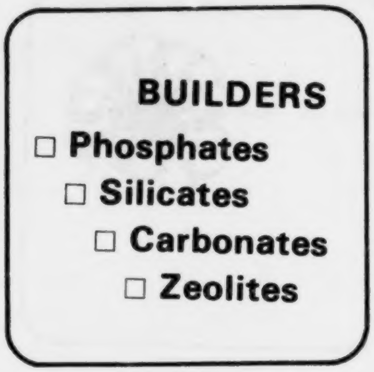
SERIES X -34, SURFACTANTS



BASIC DEFINITIONS

- DETERGENT
- SURFACE ACTIVE MATERIAL
- (SURFACTANT)
- SYNTHETHIC DETERGENT
- BUILDER

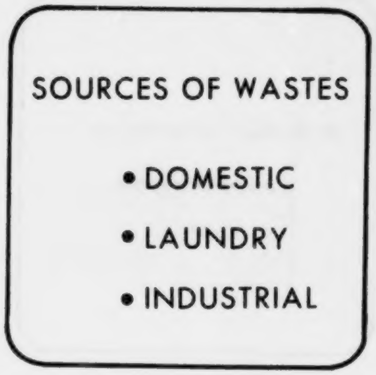
SLIDE 1



BUILDERS

☐ **Phosphates**
☐ **Silicates**
☐ **Carbonates**
☐ **Zeolites**

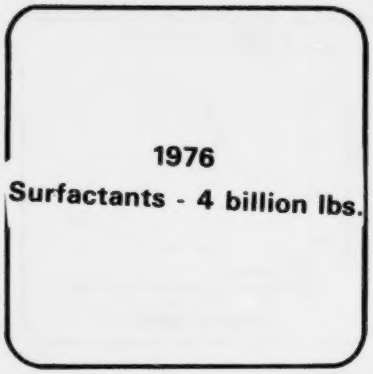
SLIDE 2



SOURCES OF WASTES

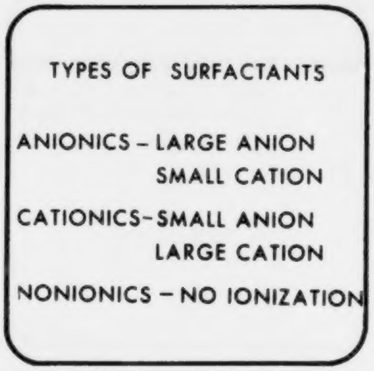
- DOMESTIC
- LAUNDRY
- INDUSTRIAL

SLIDE 3



1976
Surfactants - 4 billion lbs.

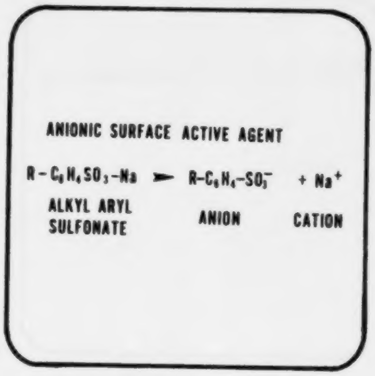
SLIDE 4



TYPES OF SURFACTANTS

ANIONICS – LARGE ANION
SMALL CATION
CATIONICS–SMALL ANION
LARGE CATION
NONIONICS – NO IONIZATION

SLIDE 5

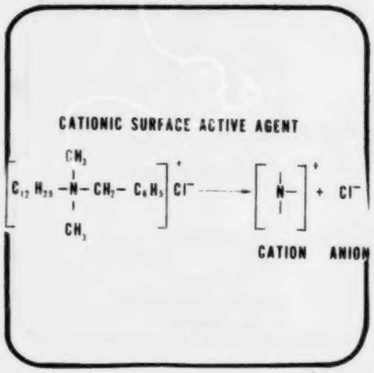


ANIONIC SURFACE ACTIVE AGENT

$R-C_6H_4SO_3-Na \rightarrow R-C_6H_4-SO_3^- + Na^+$

ALKYL ARYL SULFONATE	ANION	CATION

SLIDE 6

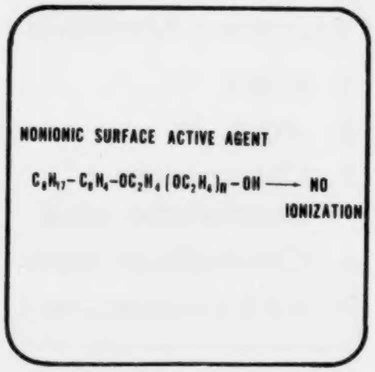


CATIONIC SURFACE ACTIVE AGENT

$$\left[C_{12}H_{25}-\overset{\overset{CH_3}{|}}{\underset{\underset{CH_3}{|}}{N}}-CH_2-C_6H_5\right]^+ Cl^- \longrightarrow \left[-\overset{|}{\underset{|}{N}}-\right]^+ + Cl^-$$

CATION **ANION**

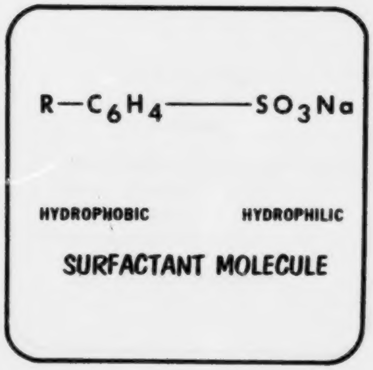
SLIDE 7



NONIONIC SURFACE ACTIVE AGENT

$C_8H_{17}-C_6H_4-OC_2H_4(OC_2H_4)_n-OH \longrightarrow$ **NO IONIZATION**

SLIDE 8

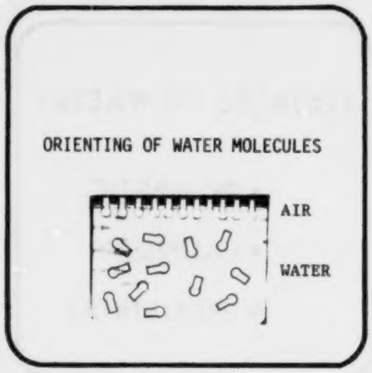


$R-C_6H_4$———SO_3Na

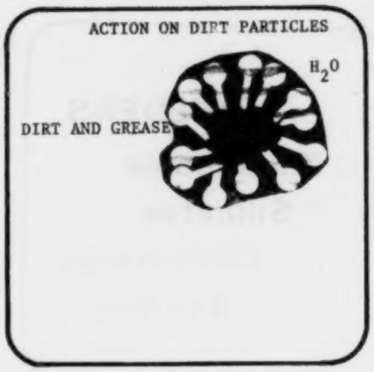
HYDROPHOBIC **HYDROPHILIC**

SURFACTANT MOLECULE

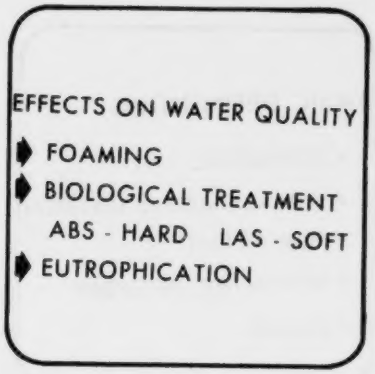
SLIDE 9


ORIENTING OF WATER MOLECULES

SLIDE 10

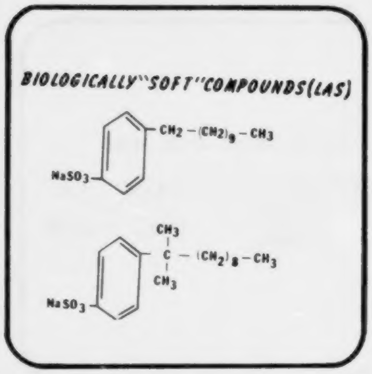

ACTION ON DIRT PARTICLES
H_2O
DIRT AND GREASE

SLIDE 11

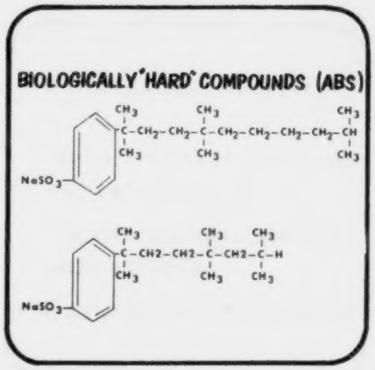

EFFECTS ON WATER QUALITY

- FOAMING
- BIOLOGICAL TREATMENT
  ABS - HARD LAS - SOFT
- EUTROPHICATION

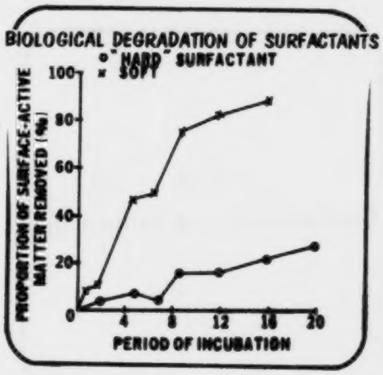
SLIDE 12


BIOLOGICALLY "SOFT" COMPOUNDS (LAS)
CH2 – (CH2)9 – CH3
NaSO3
CH3
C – (CH2)8 – CH3
CH3
NaSO3

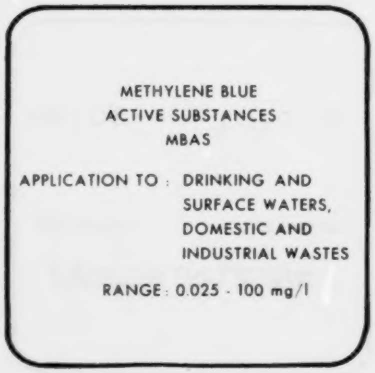
SLIDE 13


BIOLOGICALLY "HARD" COMPOUNDS (ABS)
CH3
C–CH2–CH2–C–CH2–CH2–CH2–CH2–CH
CH3
CH3
CH3
CH3
NaSO3
CH3
C–CH2–CH2–C–CH2–C–H
CH3
CH3
CH3
CH3
CH3
NaSO3

SLIDE 14


BIOLOGICAL DEGRADATION OF SURFACTANTS

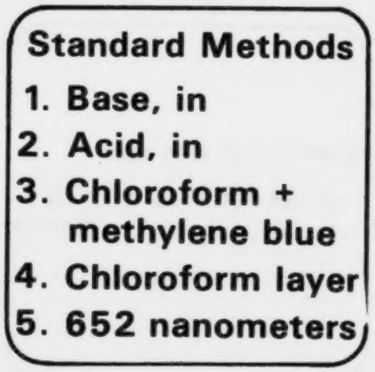
SLIDE 15


METHYLENE BLUE
ACTIVE SUBSTANCES
MBAS

APPLICATION TO : DRINKING AND
SURFACE WATERS,
DOMESTIC AND
INDUSTRIAL WASTES

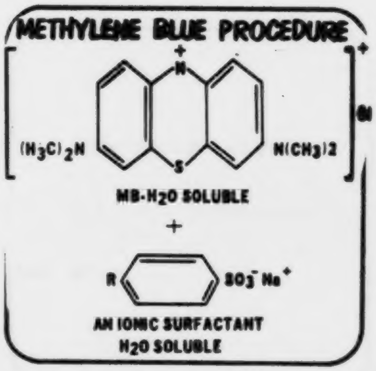
RANGE : 0.025 - 100 mg/l

SLIDE 16


Standard Methods

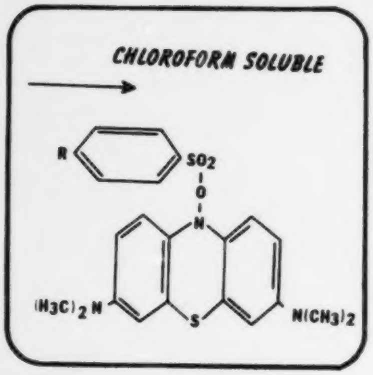
1. Base, in
2. Acid, in
3. Chloroform + methylene blue
4. Chloroform layer
5. 652 nanometers

SLIDE 17

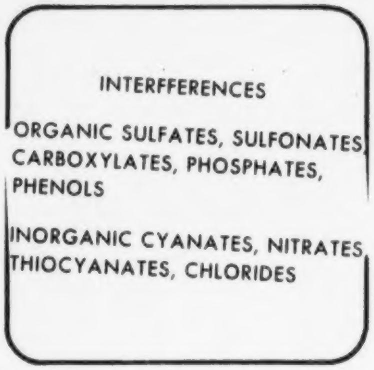

METHYLENE BLUE PROCEDURE
+
N
S
(H3C)2N
N(CH3)2
+
MB-H2O SOLUBLE
+
R
SO3⁻ Na⁺
AN IONIC SURFACTANT
H2O SOLUBLE

SLIDE 18

SERIES X -34, SURFACTANTS


CHLOROFORM SOLUBLE
R
SO2
O
N
S
(H3C)2N
N(CH3)2

SLIDE 19



INTERFFERENCES

ORGANIC SULFATES, SULFONATES, CARBOXYLATES, PHOSPHATES, PHENOLS

INORGANIC CYANATES, NITRATES, THIOCYANATES, CHLORIDES

SLIDE 20 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Introduction to Gas Chromatography

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 45 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for the determination of such organic pollutants as chlorinated hydrocarbons and polychlorinated biphenyls to meet water quality control program requirements. A knowledge of gas chromatography is needed in order to be able to carry out these, and other analyses.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of college organic chemistry, or equivalent, and understand the terms ng, μg, mg, g, ml, and l as metric units of mass and volume.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on the Subject Matter, and will know the fundamentals of gas chromatography.

B. Conditions:

He/she will have heard the lecture on the Subject Matter.

C. Accepted Performance:

1. A grade of 70% on the quiz.
2. Attentiveness during the lecture, as judged by the instructor.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in the Training Manual:
   a. Introduction to Gas-Liquid Chromatography
   b. Quantitation in Gas Chromatography
2. X-36, Gas Chromatography (5 slides); see XI, Description of Visual Materials (page 22-6).
3. An Introduction to Gas Chromatography, National Training and Operational Technology Center, Paul F. Hallbach. A copy follows page 22-6.

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.

2. Reproduce the attached quiz; one copy for each participant, plus any desired file copies. See pages 22-4 and 22-5.

3. Reproduce item VII A. 3.; one copy for each participant, plus any desired file copies.

B. Sequencing:

Classroom - discuss the Training Manual outlines, Introduction to Gas-Liquid Chromatography, and Quantitation in Gas Chromatography, using X-36, Gas Chromatography.

1. Introduction:
    a. Gas chromatography is a tool to separate and identify a mixture of volatile components in a mixture.
    b. The components are usually organic.
    c. Gas chromatography is not as specific as infrared, atomic absorption, or mass spectroscopy.
    d. Proof of component identity:
        1) Retention time
        2) Use of multiple columns
        3) Use in conjunction with other instruments
2. Parts of a gas chromatograph:
    a. Carrier gas; X-36, 1
    b. Injection port; X-36, 1
    c. Column; X-36, 1 and 2
    d. Detectors; X-36, 3 and 4
    e. Qualitative and quantitative identification; X-36, 5

Classroom - distribute copies of item VII A. 3. (Note: it is not necessary for the participants to study this document prior to taking the quiz).

Classroom - final discussion and questions.

Classroom - give the quiz and then read the correct answers from the attached answer sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

For Each Student:

Handout materials.

X. IPW REAGENT REQUIREMENTS:

None

NAME____________________________

QUIZ
Introduction to Gas Chromatography

Check the correct answer or fill in the missing word(s) as indicated.

1. Gas-liquid chromatography is an analytical method for the ____________________ and ____________________ of a mixture of volatile components in a sample.
2. Four of the components in a gas chromatograph are ____________________, ____________________, ____________________, and ____________________.
3. Three types of gas chromatographic detectors (use class names or names of specific types) are ____________________, ____________________, and____________________
4. The time required for a particular component to travel through the gas chromatographic column is called the ____________________ time.
5. Two general ways of quantitating peaks in gas chromatography are the measurement of ____________________ and ____________________.

NAME________________________

QUIZ - ANSWER SHEET

Introduction to Gas Chromatography

Check the correct answer or fill in the missing word(s) as indicated.

1. Gas-liquid chromatography is an analytical method for the separation and identification of a mixture of volatile components in a sample.

2. Four of the components in a gas chromatograph are carrier gas or gas, injector, column, and detector.

3. Three types of gas chromatographic detectors (use class names or names of specific types) are thermal conductivity, ionization, and microcoulometric; or flame ionization, or electron capture, or thermionic.

4. The time required for a particular component to travel through the gas chromatographic column is called the retention time.

5. Two general ways of quantitating peaks in gas chromatography are the measurement of peak area and peak height.

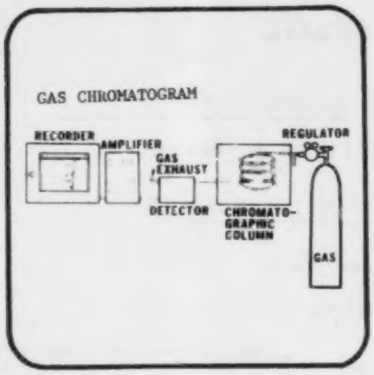
Note to Instructor:

It is suggested that one point be awarded for each correct answer.

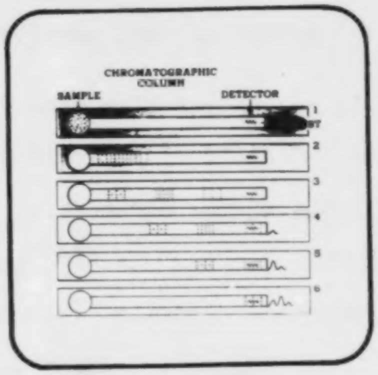
For "fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

XI. DESCRIPTION OF VISUAL MATERIALS

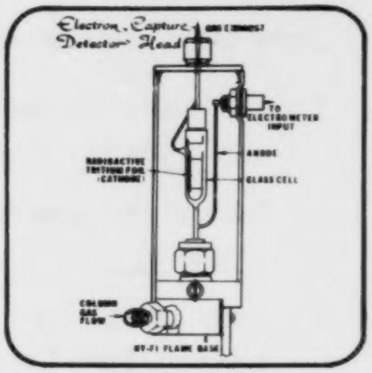
SERIES X-36, GAS CHROMATOGRAPHY


GAS CHROMATOGRAM
RECORDER
AMPLIFIER
GAS EXHAUST
REGULATOR
DETECTOR
CHROMATOGRAPHIC COLUMN
GAS

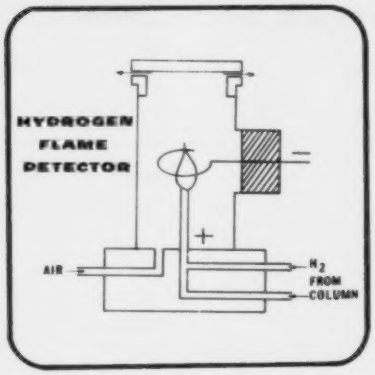
SLIDE 1


CHROMATOGRAPHIC COLUMN
SAMPLE
DETECTOR
1
DDT
2
3
4
5
6

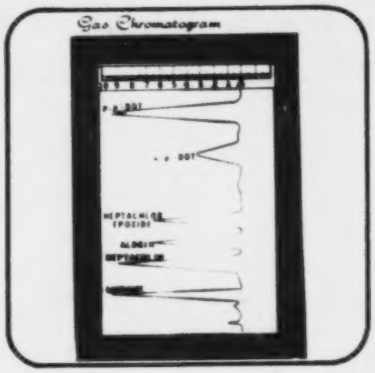
SLIDE 2


Electron Capture Detector Head
GAS EXHAUST
TO ELECTROMETER INPUT
ANODE
RADIOACTIVE TRITIUM FOIL (CATHODE)
GLASS CELL
COLUMN GAS FLOW
GY-FI FLAME BASE

SLIDE 3


HYDROGEN FLAME DETECTOR
−
+
AIR
H_2
FROM COLUMN

SLIDE 4



SLIDE 5 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

SLIDE

AN INTRODUCTION TO GAS CHROMATOGRAPHY

BY

Paul F. Hallbach
National Training Center
Water Program Operations
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

22-ba

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I INTRODUCTION 1

II CHROMATOGRAPHIC SYSTEM 2

A Carrier Gas 2

B Sample Introduction 4

C Column Materials 4

D Solid Support 4

E Stationary Phase 4

F Temperature Control 5

G Detectors 5

- 1 Thermal conductivity 6
- 2 Ionization detector 8
  - a Electron capture 11
  - b Flame ionization 11
- 3 Microcoulometric detector 12
- 4 Flame Photometric detector 12

III QUANTITATION 15

IV REFERENCES 16

22-6b

INTRODUCTION TO GAS LIQUID CHROMATOGRAPHY

OBJECTIVES: In this lesson you will learn the fundamental operating characteristics of a gas chromatograph; and you will understand how this instrument can be used as an analytical tool for the estimation of trace organic compounds in water supplies.

STUDY TIME REQUIRED: 4 hours.

I INTRODUCTION

The analytical chemist is frequently confronted with the problem of separating various compounds, which can be present in a sample, prior to analysis. In 1903, Tswett(1) discovered that he could separate plant pigments into discrete colored bands. He dissolved a mixture of plant pigments (chlorophylls) in petroleum ether and passed the solution through a column of calcium carbonate. He noticed that yellow and green zones were formed in the column. As a result of his experiment he coined the term "chromatography", which translated literally means "color writing". This term is now applied almost universally to all forms of physical separations of compounds.

In the early forties Martin and Synge(2) were disappointed with methods of separation based on differences of partitioning coefficients of various compounds in solvents and the use of separatory funnels. They discovered that separation of nucleic acids could be affected by using a column of silica gel containing definite amounts of water. The compounds were adsorbed onto the top section of the column as in Tswett chromatography and then removed separately with an organic solvent. In this case, the column contained silica gel and a stationary phase of water. The mobile organic phase transported the compounds over the "stationary" aqueous phase. In this way the transported compounds were partitioned between the two phases. It was necessary for the compounds to have some solubility in water. Since their partition coefficients differed sufficiently each compound traveled for differing lengths of time in the column and were therfore separated from each other.

22-6c

In 1952 James and Martin(3) substituted a gas as the mobile organic phase and published their classical paper describing the gas-liquid partition technique and a relatively simple apparatus for the separation of aliphatic acids. The solutes were distributed between a moving carrier gas, nitrogen, and a nonvolatile liquid phase, silicone oil containing some stearic acid, immobilized on a finely divided solid, Celite. The potentialities of the method became apparent to chemists almost immediately, and intensive research on gas chromatography was carried out in laboratories throughout the world. Subsequently, a new literature has developed which is devoted to this technique and now includes more than four thousand publications.

II CHROMATOGRAPHIC SYSTEM

One of the characteristics of Gas Chromatography is the simplicity of the apparatus requirements for using the technique in a wide range of separation problems. The most refined equipment contains four basic components optimized in design to realize the potential advantages of efficiency, sensitivity and convenience.

The apparatus for any Gas Chromatographic separation (Figure 1) consists of:

1. A carrier gas supply and flow control
2. A sample port and a means of introducing the sample
3. A chromatographic column and temperature control conducive to maintaining the sample in a volatilized state
4. A detector

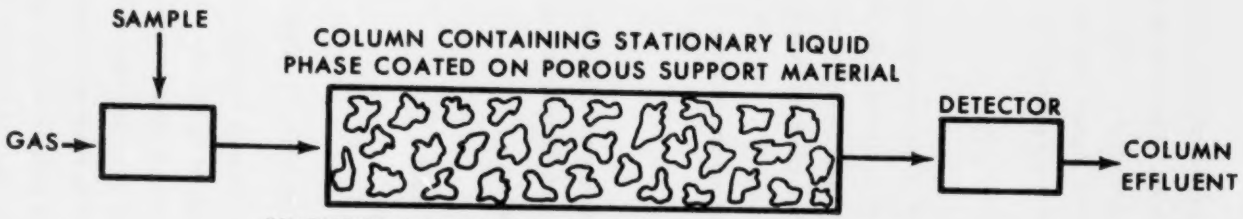
A Carrier Gas

A high pressure gas cylinder serves as the source of carrier gas. A pressure regulator is used to assure a uniform pressure to the column inlet, and thereby a constant rate of gas flow. At a given temperature this constant rate of flow will elute components at a characteristic time (the retention time). Since the flow rate is constant, the components also have a characteristic volume of carrier gas (the retention volume).

Commonly used gases are hydrogen, helium and nitrogen. The carrier gas should be:

1. Inert to avoid interaction with the sample or solvent

22-6d


SAMPLE
GAS
COLUMN CONTAINING STATIONARY LIQUID PHASE COATED ON POROUS SUPPORT MATERIAL
DETECTOR
COLUMN EFFLUENT

SIMPLIFIED SCHEMATIC OF GAS CHROMATOGRAPH

Figure 1

22-6e

2 Provide a minimum of gaseous diffusion
3 Readily available and of high purity
4 Inexpensive

B Sample Introduction

Liquid samples are injected into the injection port of the instrument with a micro-liter syringe. The volume most generally used is in the order of 5 to 50 micro-liters. The temperature of the injection port is generally 25-40° C higher than the column so that the sample is volatilized instantaneously prior to its travel through the chromatographic column.

C Column Materials

The column is the heart of the chromatograph. The actual separation of sample components is achieved in the column. In a gas chromatograph, the solid support material and stationary liquid phase (column packing) is contained in copper, stainless steel, aluminum or glass tubing. The tubing is usually 1/4 to 1/8 inch in diameter, and 5 to 10 feet in length. Columns are coiled to fit the temperature controlled oven of the instrument.

D Solid Support

The purpose of the solid support is to provide a large, uniform, inert surface area for distributing the stationary liquid phase. Desirable properties are:

1 Inert
2 High crushing strength
3 Large surface area
4 Uniform size

The most widely used material is calcined diatomaceous earth.

E Stationary Phase (Partitioning Solvent)

The stationary liquid phase is usually a material similar to silicone oil or grease. In the actual preparation of the column, the liquid phase is slurried in a solvent such as ethylacetate and poured over the support material. The solvent is removed by evaporation and a thin coating of the partitioning liquid remains on the surface of the support material. Various techniques are used to provide uniform coating of the support material.

22-6f

The correct choice of the partitioning solvent is probably the most important parameter in Gas-Liquid Chromatography (GLC). Ideally the solvent should have the following characteristics:

1 Samples must exhibit different distribution coefficients.
2 Samples should have a reasonable solubility in the solvent.
3 The solvent should have a negligible vapor pressure at operating temperature.

For an efficient, normal operation, the liquid phase should be similar in chemical structure to the components of the mixture. Considerable column technology has been reported in the literature. The selection of the liquid phase and method of coating can easily be made after an investigation of appropriate literature references.

F Temperature Control

The instrument should provide separate temperature controls for the injection port, the oven containing the column, and the detector. The injection port should be hot enough to vaporize the sample so rapidly that no loss in efficiency results from the injection technique. The column temperature should be high enough that the analysis is accomplished in a reasonable length of time, and low enough that the desired separation is obtained. Retention times double for every 30° C decrease in column temperature. Obviously the temperature of the column should be such that the stationary liquid phase will not be volatilized.

G Detectors

The detector indicates the presence and measures the amount of components in the column effluent. James and Martin, in their work previously described, simply bubbled the gas effluent from the column into separate aqueous solutions and titrated the acidic solutions with standard alkali. The individual compounds were separated, since their respective retention times in the column were different, and a quantitative estimation could easily be obtained. Desirable characteristics of a detector are: high sensitivity, low noise level, wide linearity of response, ruggedness, response to compounds of interest, insensitive to gas flow and temperature changes, inexpensive.

22-6g

There is no ideal detector and therefore many detectors have been devised for specific applications. The detectors which are of primary interest to the water chemist are:

1 Thermal conductivity

Obviously more sensitive detectors were needed. The thermal conductivity detector is based on the principle that a hot object will lose heat at a rate which is dependent upon the composition of the surrounding gas. In this detector, two wires are positioned independently and heated to the same temperature electrically. Since the amount of electricity which flows through the wire is temperature dependent we have the basis for a suitable detector. If pure carrier gas is used as the composition of the surrounding gas for one wire and the column effluent is used as the surrounding gas for the other wire, then there will be a difference in the amount of current flowing when an impurity (sample component) is present in the column effluent.

The principle upon which this detector is based was reported as the basis for determining the purity of a gas stream. The device was patented in 1915 by Shakespear and called a "katharometer" (from the Greek word "katharos" meaning pure). The thermal conductivity (TC) detector was introduced into gas chromatography by Claesson(4) in 1946. Sensitivity of the detector was subsequently increased by the use of thermistors. Since the absolute measurement of thermal conductivity is very difficult, the differential technique is generally used. The resistance element is used as a matched pair, one exposed to the carrier gas and the other to the column effluent in a cell such as shown in Figure 2. The reference and sensing resistance elements are incorporated into a Wheatstone bridge, and the out-of-balance signal is applied to a recorder.

The principal application in relation to water pollution control for the detector has been for the separation and analysis of anaerobic digester gases.

The use of this detector is somewhat limited in its application because it lacks the sensitivity that can be obtained with ionization type detectors.

22-64

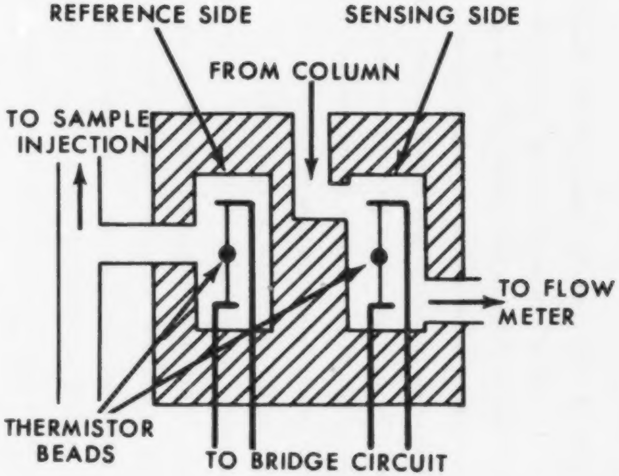

REFERENCE SIDE
SENSING SIDE
FROM COLUMN
TO SAMPLE INJECTION
TO FLOW METER
THERMISTOR BEADS
TO BRIDGE CIRCUIT

Figure 2 THERMAL CONDUCTIVITY CELL

22-6I

2 Ionization detectors

The fundamental physical process upon which the operation of ionization detectors operates is the conduction of electricity by gases. At normal temperatures and pressures a gas behaves as a perfect insulator. If electrically charged atoms, molecules, or free electrons are present, their free motion in the direction of an electrical field renders the gas conducting. In the absence of conduction by the gas molecules themselves, the increased conductivity due to the presence of very few charged molecules can be observed and this explains the great sensitivity of ionization methods for gas analysis.

The conduction of electricity by gases can be observed by reference to Figure 3. It consists of an insulating vessel through which gas can be passed and which has two conductors supported within it. The vessel contains some source of energy capable of splitting the otherwise electrically neutral gas molecules into ion pairs -- for example, a flame or a source of ionizing radiation. The electrical circuit external to the chamber is completed through an apparatus for the measurement of current flow to a source of electrical potential. A typical schematic ionization detector circuit is shown in Figure 4. Effluent gas from the column flows through the electrode gap past an ionizing source which ionizes some of the molecules in the gas stream. The presence of charged particles (positive ions, negative ions, electrons) within the electrode gap causes a current to flow across the gap and through a measuring resistor R_2. The resulting voltage drop E_0 across R_2 is amplified by an electrometer and fed into a recorder.

At least nine different detector systems have been developed for specific use in gas chromatography which utilize this principle. They all exploit some single property of a gaseous conductor for detection purposes. Two ionization detectors which are most widely used by water chemists are the electron capture and the flame ionization detectors.

4 22-65

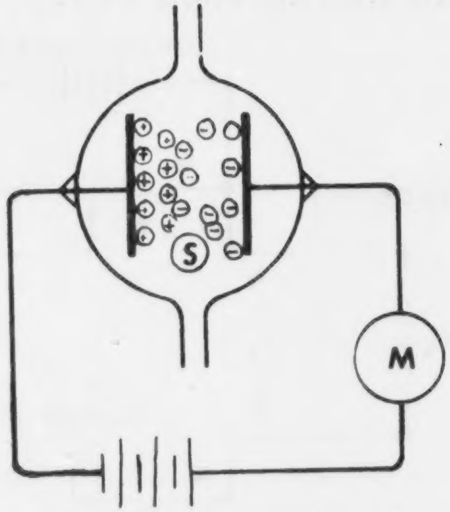


Figure 3 A SIMPLE IONIZATION CHAMBER

S- Source of ions
M- Apparatus for measuring current

22-6K

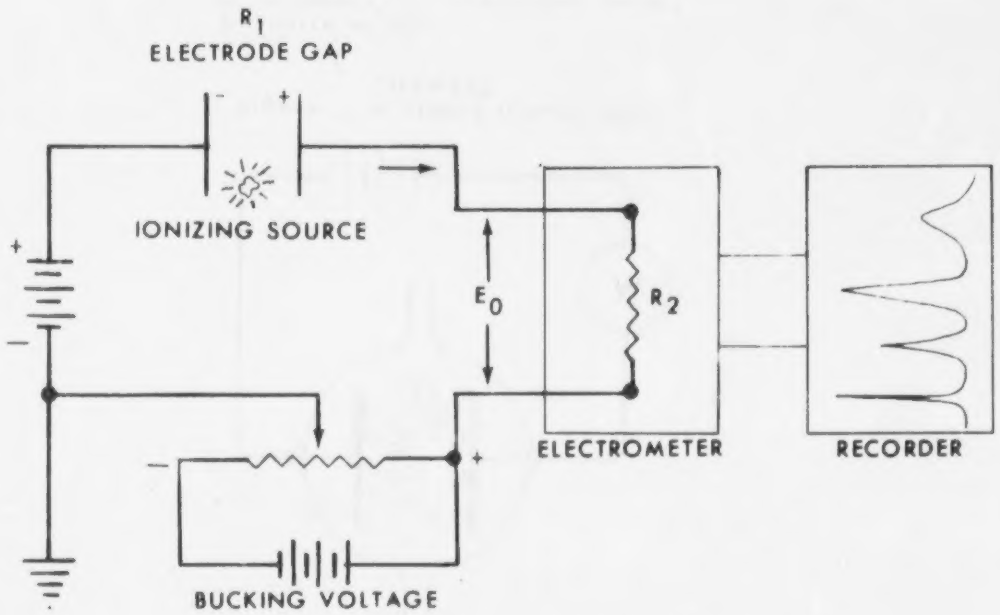

R1
ELECTRODE GAP
-
+
IONIZING SOURCE
+
-
E0
R2
ELECTROMETER
RECORDER
-
+
BUCKING VOLTAGE

Figure 4 IONIZATION DETECTOR CIRCUIT

22-6L

a Electron capture detector

This detector schematically shown in Figure 5 was specifically designed for the measurement of compounds having an affinity for free electrons. A radioactive source such as tritium is utilized to provide energy for the ionization of the carrier gas. Secondary electrons are produced and an electrical current flows between the electrodes in the detector. When a separated compound which has an affinity for the slow electrons enters the cell, electrons are captured with a resultant decrease in electrical current in the detector. This decrease of current is a function of the concentration of the electron capturing compound.

For quantitative analysis this detector is best suited to measurements at high sensitivity of organic and inorganic halogen-containing compounds and also certain oxygen-containing compounds such as volatile nitrates, ozone and oxygen. Selective sensitivity to halides makes this detector especially valuable for the analysis of pesticides.

The detector is capable of quantitating concentrations as low as 10^{-12} grams (picogram).

b Flame ionization detector

In this detector a flame is used as a source of energy for splitting the otherwise electrically neutral gas molecules into ion pairs. A schematic diagram of a flame-ionization cell is shown in Figure 6. The column effluent is mixed with hydrogen fuel and burned at the tip of the metal jet in an excess of air. Hydrogen burning at the jet produces a very low background flow of current of the order of 10^{-14} amperes which can be electrically compensated. When an organic compound which has been separated in the column, enters the cell, an increase in ions is produced in the flame and the amount of current flowing in the cell is also increased. The magnitude of the current is a measure of the concentration of the unknown compound. The detector is

22-6m

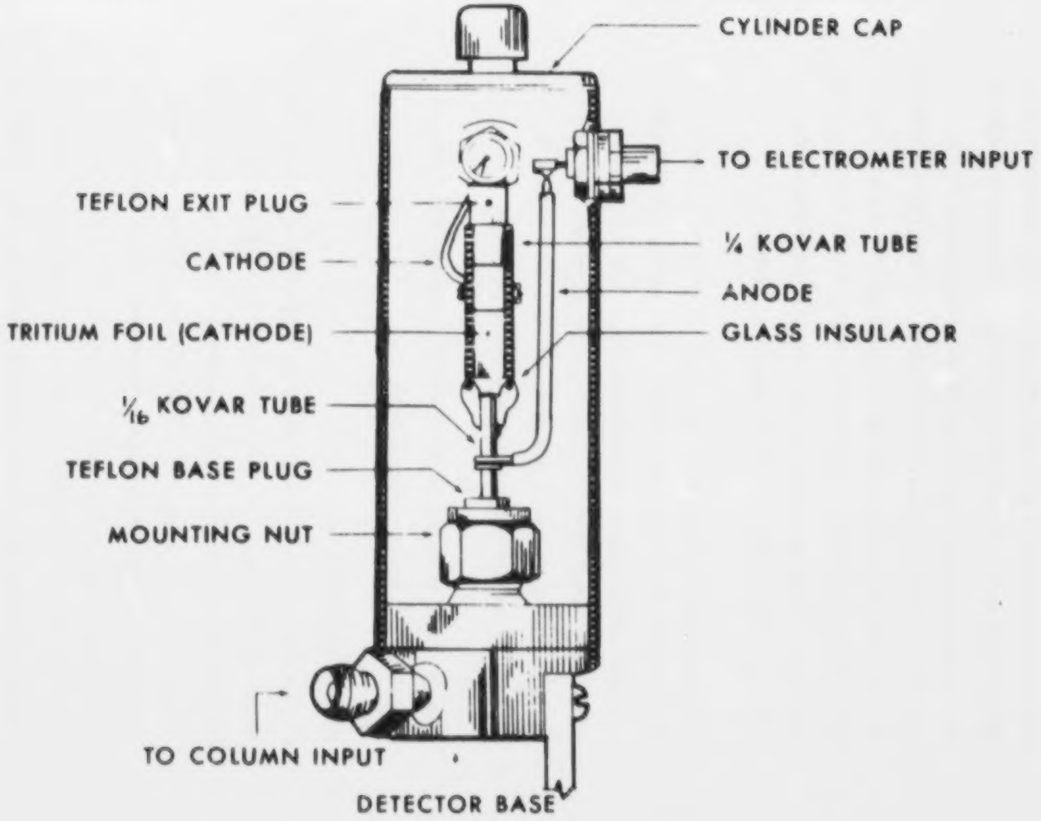

CYLINDER CAP
TO ELECTROMETER INPUT
TEFLON EXIT PLUG
CATHODE
¼ KOVAR TUBE
ANODE
TRITIUM FOIL (CATHODE)
GLASS INSULATOR
1/16 KOVAR TUBE
TEFLON BASE PLUG
MOUNTING NUT
TO COLUMN INPUT
DETECTOR BASE

Figure 5 ELECTRON CAPTURE DETECTOR

22-6n

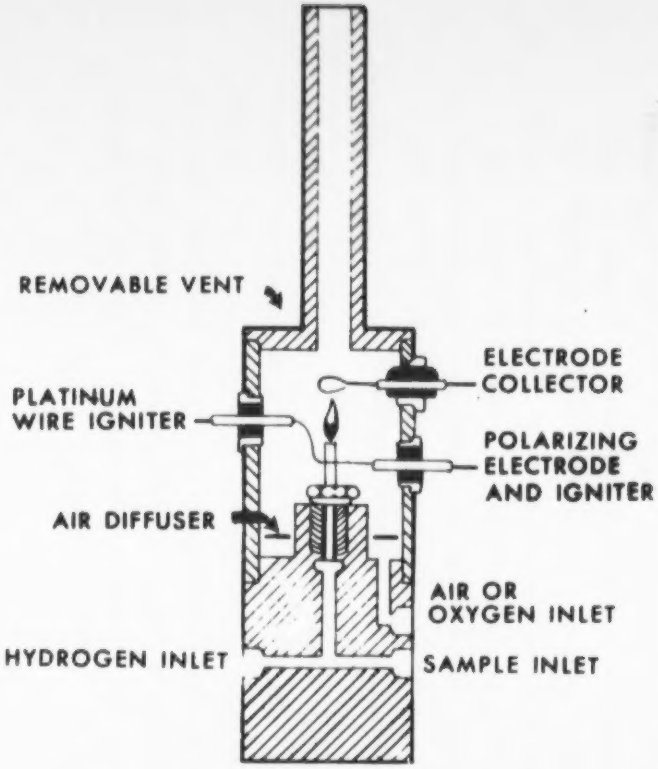

REMOVABLE VENT
ELECTRODE
COLLECTOR
PLATINUM
WIRE IGNITER
POLARIZING
ELECTRODE
AND IGNITER
AIR DIFFUSER
AIR OR
OXYGEN INLET
HYDROGEN INLET
SAMPLE INLET

Figure 6 FLAME IONIZATION CELL

22-60

sensitive to all compounds containing carbon with the exception of carbon disulfide, carbon monoxide and carbon dioxide. The detector has been modified by the addition of an alkali salt to the collecting electrode to provide increased sensitivity to organic compounds containing phosphorus. The modified detector is called "thermionic" and is used for organo-phosphate pesticide analyses.

3 Microcoulometric detector

This detector consists of titration cells for the measurement of chloride-containing and sulfur-containing compounds. Schematically it is shown in Figure 7. The sample component emerging from the column is combusted to produce hydrogen chloride or sulfur dioxide, respectively. HCl is continuously titrated by silver ions present in the cell; the amount of current required to regenerate these silver ions is recorded. The system for sulfur-containing compounds is analogous except that SO_2 is continuously titrated by iodine which is subsequently regenerated. This detector has had wide use in pesticide analysis.

4 Flame photometric detector

Recently a flame photometric detector was introduced. Its operation is based on the principles of flame emission spectroscopy. A conventional flame ionization cell is used and a dual read-out of the flame emission and flame ionization response can be recorded. The detector has found wide acceptance for the analysis of organo-phosphate pesticides.

III QUALITATIVE AND QUANTITATIVE ANALYSES

The most extensive applications for the technique of gas chromatographic analysis has been in the separation of complex mixtures and in the qualitative and quantitative information which may be obtained through interpretation of the recorded electrical response (chromatogram).

The classical approach to qualitative analysis by gas chromatography is based on a comparison of the retention time or volume of the unknown

22-60

MICROCOULOMETRIC GAS CHROMATOGRAPH

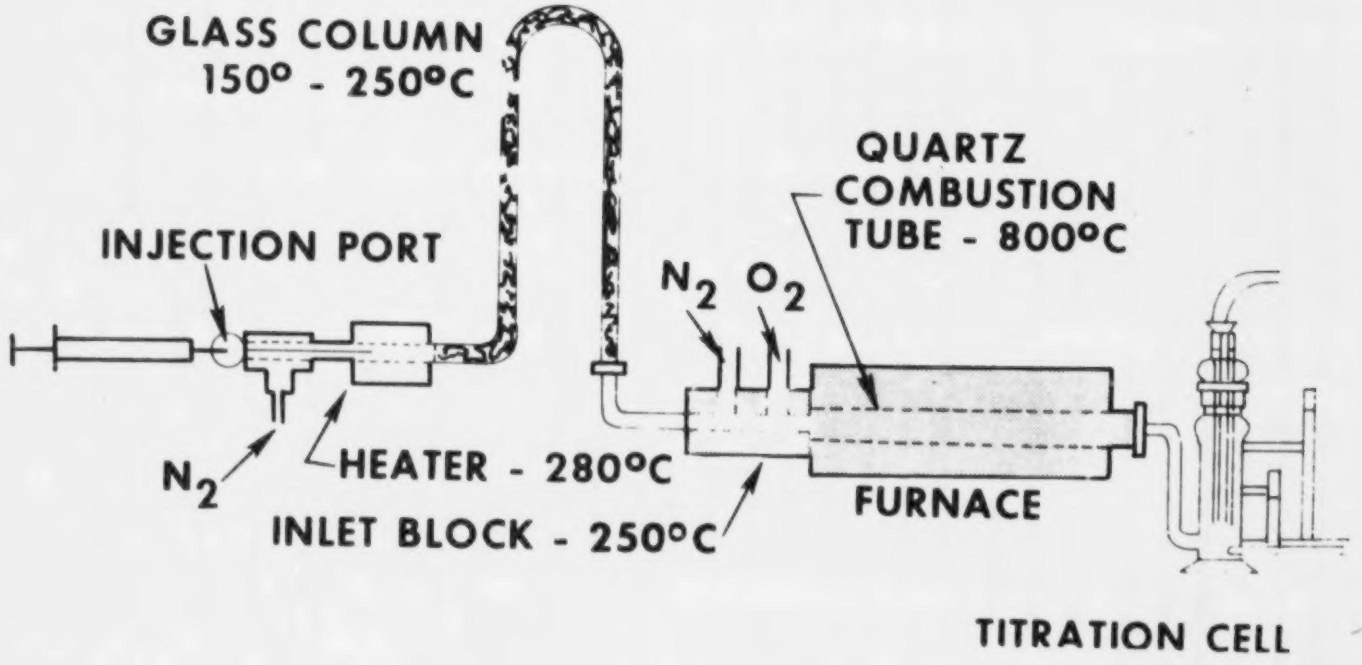

GLASS COLUMN
150° - 250°C
INJECTION PORT
N_2
HEATER - 280°C
INLET BLOCK - 250°C
N_2 O_2
QUARTZ
COMBUSTION
TUBE - 800°C
FURNACE
TITRATION CELL

Figure 7

22-6p

component with that obtained for a known compound run on the same column under identical operating conditions.

Quantitative measurements using thermal conductivity cells, ionization detectors, or other differential type detectors depend upon the determination of the recorded electrical response area (peak) or peak height and the relationship of these quantities to the amount or concentration of solute in the sample. Suitable calibration curves can thus be prepared by relating peak height or area of solutions of known concentrations.

A Methods of Peak Area Measurements

Area measurements are commonly carried out by one of the following techniques:

1 Planimetering. The peak is traced manually with a planimeter. A planimeter is a mechanical device which measures area by tracing the perimeter of the peak. The area is presented digitally on a dial.

2 Height × Width at Half-Height. Since normal peaks approximate a triangle, one could approximate the area by multiplying the peak height times the width at half-height. This technique is rapid and simple, and is illustrated in Figure 8.

3 Triangulation. Height of the peak is measured from the baseline to the intersection of the two tangents (Figure 9). The base is taken as the intersection of the two tangents with the baseline. The area is calculated by the triangle formula: area = 1/2 BH.

REFERENCES

1 Tswett, M. Proc. Warsaw Co. Nat. Sci., Biol. Sec. 14, Minute No. 6 (1903).

2 Martin, A.J.P. and Synge, R.L.M. Biochem. J. 35:1358 (1941).

3 James, A.T. and Martin, A.J.P. Biochem. J. 50:679 (1952).

4 Claesson, S. Ark. Kemi. Min. Geol. A-23, No. 1:133 (1946).

5 Dal Nogare, S. and Juvet, R.S. Jr. Gas Liquid Chromatography. New York, Interscience. (1962).

6 McNair, H.M. and Bonelli, E.J. Basic Gas Chromatography. Varian Aerograph Corp. (1967).

22-bq

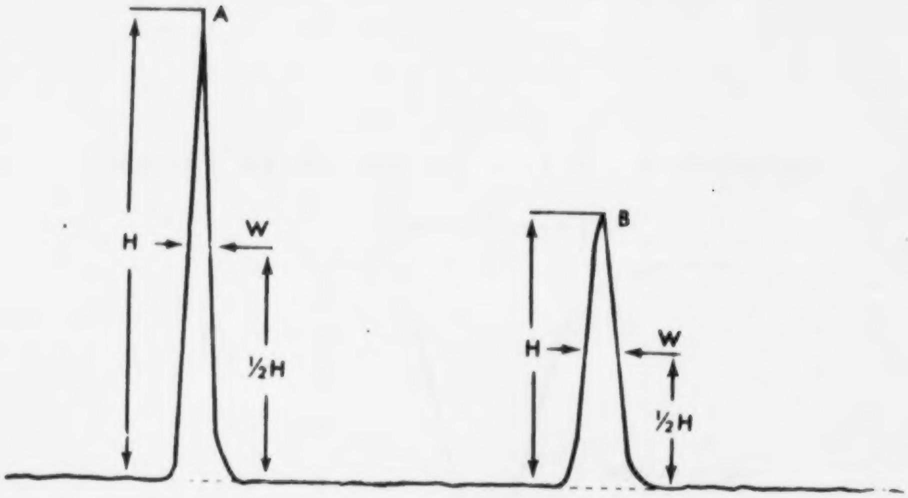

A
H
W
½H
B
H
W
½H

Figure 8 PEAK HEIGHT MEASUREMENT BY HxW

22-6R

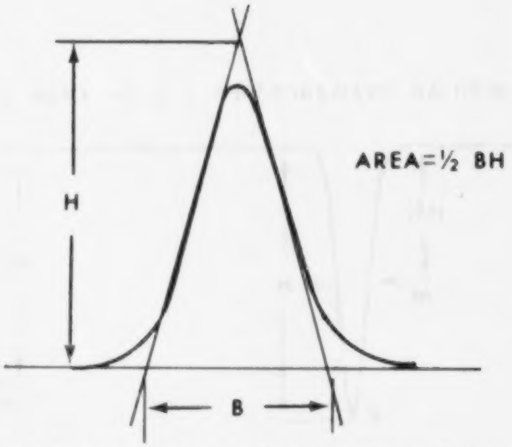


Figure 9 MEASUREMENT OF AREA BY TRIANGULATION

22.65

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Extraction of Chlorinated Hydrocarbons

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 2 hours, 10 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of college organic chemistry, or equivalent, and have the ability to:

A. Handle organic solvents safely.

B. Understand the terms ng, µg, mg, g, ml, and l as metric units of mass and volume.

C. Use a separatory funnel to perform liquid-liquid extractions.

D. Perform simple calculations (addition, subtraction, multiplication, division) involving decimals.

E. Prepare a calibration graph and use it to determine the concentration of a sample constituent.

F. Use ordinary laboratory glassware (beakers, flasks, graduated cylinders, graduated pipets, volumetric pipets, etc.)

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will extract a mixture of lindane, aldrin and heptachlor expoxide from a distilled water sample, inject the mixture into a gas chromatograph, prepare a calibration graph, and use it to determine the concentration of each of the three components in the sample.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual and the equipment/supplies/reagents listed in sections IX and X of this IPW.

C. Accepted Performance:

1. Value for each of the three components within ± 10% of the class average.

2. The instructor will judge whether or not the calibration graph has been prepared properly (e.g., use of the entire x and y axes, and line of best fit).

3. The instructor will judge whether or not proper injection technique has been used (e.g., no contamination of needle or plunger, accurate volume measurement, no rough handling of the syringe).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

One outline in the Training Manual:

Method for Organochlorine Pesticides in Industrial Effluents (according to procedure by US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
2. Review the laboratory procedure by performing the student assignment.
3. Prepare the equipment/supplies/reagents listed in Sections IX and X of this IPW.
4. Muffle beakers of anhydrous sodium sulfate at 400°C for 4 hours, (Approximately 42 g per beaker; 1 beaker for each laboratory station).
5. Just before the laboratory session, "spike" 500 ml of distilled water in a separatory funnel at each station with 3 ml of a mixture of lindane, aldrin, and heptachlor epoxide in hexane. The concentration of each of the 3 components in the "spike" mixture should be 0.33 ng/μl. Pure pesticide standards may be ordered from: US EPA, EMSL, Quality Assurance Branch, Cincinnati, Ohio 45268.

   $$3 \text{ ml } (3000\ \mu\text{l}) \times 0.33\ \frac{\text{ng}}{\mu\text{l}} = 990 \text{ ng} \approx 1000 \text{ ng}$$

   1000 ng of each of the 3 components in 500 ml = 2.0 μg/l of sample. In the "final" 10 ml of extracting solvent, the spike concentration will be 0.1 ng/μl, the same concentration as the "middle" standard.
6. Check the gas chromatographs for proper functioning. Inject standards so as to determine proper volumes to inject and attenuation to be used by the participants. Standards and sample should be injected at the same attenuation.

B. Sequencing:

1. Laboratory - have the participants prepare a 3-4 inch column of anhydrous sodium sulfate. (See Section IX B of the Training Manual outline).

2. Laboratory - instruct the participants in the use of the Kuderna-Danish apparatus.

3. Laboratory - have the participants do sections IX A, B, and C in the Training Manual outline.

   Note: While the solvent is concentrating in the hot water bath, the participants should begin injecting standards into the gas chromatograph and preparing the calibration graph. A second staff member must monitor the solvent concentration. Inform the participants that lindane aldrin, heptachlor epoxide is the order of column elution, what volumes of standards should be injected, and what attenuation should be used.

4. Laboratory - have the participants inject the sample into the gas chromatograph.

5. Classroom - have the participants use the calibration graph to determine the concentration of each of the 3 components in μg/liter.

6. Classroom - final discussion and questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 1 sample

A. For Each Station

1 pair safety glasses (for each person at the station)

1 laboratory apron or coat (for each person at the station)

1 distilled water plastic squeeze bottle

1 grease pencil (or other marking pencil)

paper towels or sponge

1 1 liter separatory funnel with Teflon stopcock

Ring stand, ring, clamp, rack, etc., to support the separatory funnel.

1 100 ml graduated cylinder

1 1 liter Erlenmeyer flask

1 100 ml beaker

1 Chromatographic pyrex column (approximately 400 mm long by 20 mm internal diameter) with coarse fritted plate and Teflon stopcock

1 clamp

1 Kuderna-Danish (K-D) apparatus (Kontes) consisting of:
  1 500 ml evaporative flask
  1 3-ball Snyder column

1 medicine dropper

1 10 ml graduated receiver ampul with stopper

1 set (2) springs to attach the ampul to the flask

1 ring stand and clamp to support the K-D apparatus

1 powder funnel

B. Shared:

1. Desiccator(s) to hold one 150 ml beaker containing approximately 42 g anhydrous sodium sulfate for each laboratory station.
2. Muffle oven to heat the beakers of sodium sulfate at 400°C.
3. Steam bath to accomodate 1 K-D apparatus for each laboratory station.
4. Gas chromatograph; 1 per 2 stations with:
   a. glass lined injection port
   b. electron capture detector (nickel 63 or tritium)
   c. column
      1) pyrex (180 cm long by 4 mm internal diameter)
      2) solid support-gas chrom Q (100-200 mesh)
      3) liquid phase - one of the following with % coated on solid support as shown.
         a) OV-1, 3%
         b) OV-210, 5%
         c) OV-17, 1.5% + OV-210, 1.95%
         d) OV-210, 6% + SE-30, 4%
      4) glass wool-silanized
   d. rotometers - for monitoring carrier gas flow
5. Recorder; 1 per gas chromatograph
   a. potentiometric strip chart (10 in.); compatible with detector
   b. chart paper for recorder
6. Carrier gas; one each of a, b, and c per 2 gas chromatographs
   a. argon-methane (95%-5%) for pulsed detector, or nitrogen (purified grade) for direct current

   b. pressure regulator; two stage CGA 580 for nitrogen, or two stage CGA 350 for argon-methane

   c. filter for carrier gas; with

      1) oxygen trap

      2) water trap

7. Micro syringes (5, 10, and 50 µl); 1 set per gas chromatograph

8. Stopwatch (or equivalent); 1 per gas chromatograph

9. Ruler; 12 inches or 30 cm, 1 per gas chromatograph

10. Graph paper, linear; 1 per gas chromatograph

11. Tissues; several per gas chromatograph

X. IPW REAGENT REQUIREMENTS:

Minimum Amounts
(Prepare excess reagent to allow for waste, rinsing of glassware, and any desired re-runs.)

1. 200 ml of 15% methylene chloride (nanograde, distilled in glass) in n-hexane (nanograde, distilled in glass)

2. 3 pesticide (reference grade) standards mixture (in hexane); about 5 ml in small glass stoppered bottles at each gas chromatohraph.

   a. 0.05 ng lindane/µl
      0.05 ng aldrin/µl
      0.05 ng heptachlor epoxide/µl

   b. 0.10 ng lindane/µl
      0.10 ng aldrin/µl
      0.10 ng heptachlor epoxide/µl

   c. 0.15 ng lindane/µl
      0.15 ng aldrin/µl
      0.15 ng heptachlor epoxide/µl

3. sodium sulfate, anhydrous, granular, ACS; muffled at 400°C for 4 hours and stored in a 150 ml beaker in a desiccator; 1 beaker per laboratory station

4. hexane (nanograde, distilled in glass) for rinsing syringes; about 10 ml in a small glass stoppered bottle at each gas chromatograph

5. 500 ml distilled water in a 1 liter separatory funnel with Teflon stopcock at each laboratory station, "spiked" as indicated in section VIII A. 5.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Oil and Grease

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 2 hours, 50 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of college organic chemistry, or equivalent, and have the ability to:

A. Handle organic solvents safely.

B. Handle solutions of acids safely.

C. Perform weighings on an analytical balance.

D. Perform simple calculations (addition, subtraction, multiplication, division).

E. Understand the terms µg, mg, g, ml, and l as metric units of mass and volume.

F. Use a separatory funnel to perform liquid-liquid extractions.

G. Use ordinary laboratory glassware (beakers, flasks, graduated cylinders, graduated pipets, volumetric pipets, etc.)

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on the Subject Matter, and will determine the concentration of oil/grease in a sample.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual, and the equipment/supplies/reagents listed in sections IX and X of this IPW.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. Value for oil/grease concentration of sample within ± 10% of class average.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in the Training Manual:
    a. Oil and Grease
    b. Laboratory Determination of Oil and Grease (according to Methods for Chemical Analysis of Water and Wastes, US EPA, Environmental Monitoring and Support Laboratory, 1974)
2. XT-56, Determination of Grease and Oil (54 slides, 15 minute tape, script); See XI, Description of Visual Materials (page 24-8, ff).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
2. Review the laboratory procedure by performing the student assignment.
3. Reproduce the attached quiz; one copy for each participant, plus andy desired file copies. See page 24-6 and 24-7.
4. Prepare the equipment/supplies/reagents listed in Section IX and X of this IPW.
5. Obtain a sample of municipal wastewater treatment plant effluent. A maximum of 1 l per laboratory station will be needed.

B. Sequencing:

1. Classroom - discuss oil and grease using XT-56, Determination of Grease and Oil.
    a. Introduction; XT-56, 1-2.
    b. Topics to be covered; XT-56, 3.
    c. Summary of the determination; XT-56, 4.
    d. Defining terms; XT-56, 5-8.
    e. Problems caused by grease and oil; XT-56, 9-14.
    f. Sample collection and preservation; XT-56, 15-18.
    g. Extraction of sample; XT-56, 19-39.
    h. Removal of solvent; XT-56, 40-43.
    i. Calculations; XT-56, 44-49.
    j. Summary and credits; XT-56, 50-52.

2. Laboratory - have the participants do section II of the Training Manual outline, Laboratory Determination of Oil and Grease.

3. Classroom - have the participants do section III of the same outline.

4. Classroom - final discussion and questions.

5. Classroom - give the quiz and then read the correct answers from the attached answer sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 1 sample

A. For Each Station:

1 pair safety glasses (for each person at the station)

1 laboratory apron or coat (for each person at the station)

1 distilled water plastic squeeze bottle

paper towels or sponge

1 1 l graduated cylinder

1 50 ml graduated cylinder

1 10 ml graduated cylinder

1 1 l separatory funnel with Teflon Stopcock

Ring stand, ring, clamp, rack, etc., to support the separatory funnel

1 crucible tongs (Use these or tissues to handle the flask.)

2 pieces pH sensitive paper (for use at pH 2)

1 small filtering funnel, about 5 cm in diameter

1 piece medium porosity filter paper (for use with the filtering funnel)

1 suction device with pressure tubing (see diagram on page 24-5

1 125 ml distilling flask (Although the organic extracting solvent may be evaporated, it should be recovered by distillation at 70°C. This would necessitate additional equipment such as a condenser, connecting tube, ring stand, clamp, and receiving vessel).

1 clamp

1 glass rod (about 6 inches long)

B. Shared:

Analytical balance; 1 per 4 stations

Desiccator with 1 small beaker (25 or 50 ml) containing 1 g anhydrous sodium sulfate; 1 beaker per station.

Desiccator; large enough to hold all the distilling flasks. (May be the same one as above.)

Hot water bath for use at 70°C and 80°C. All the flasks must be heated at 70°C, and then at 80°C. Both heatings must be in or near a hood.

X. IPW REAGENT REQUIREMENTS:

Minimum Amounts for Each Station
(Prepare excess reagent to allow for waste, rinsing of glassware, and any desired re-runs.)

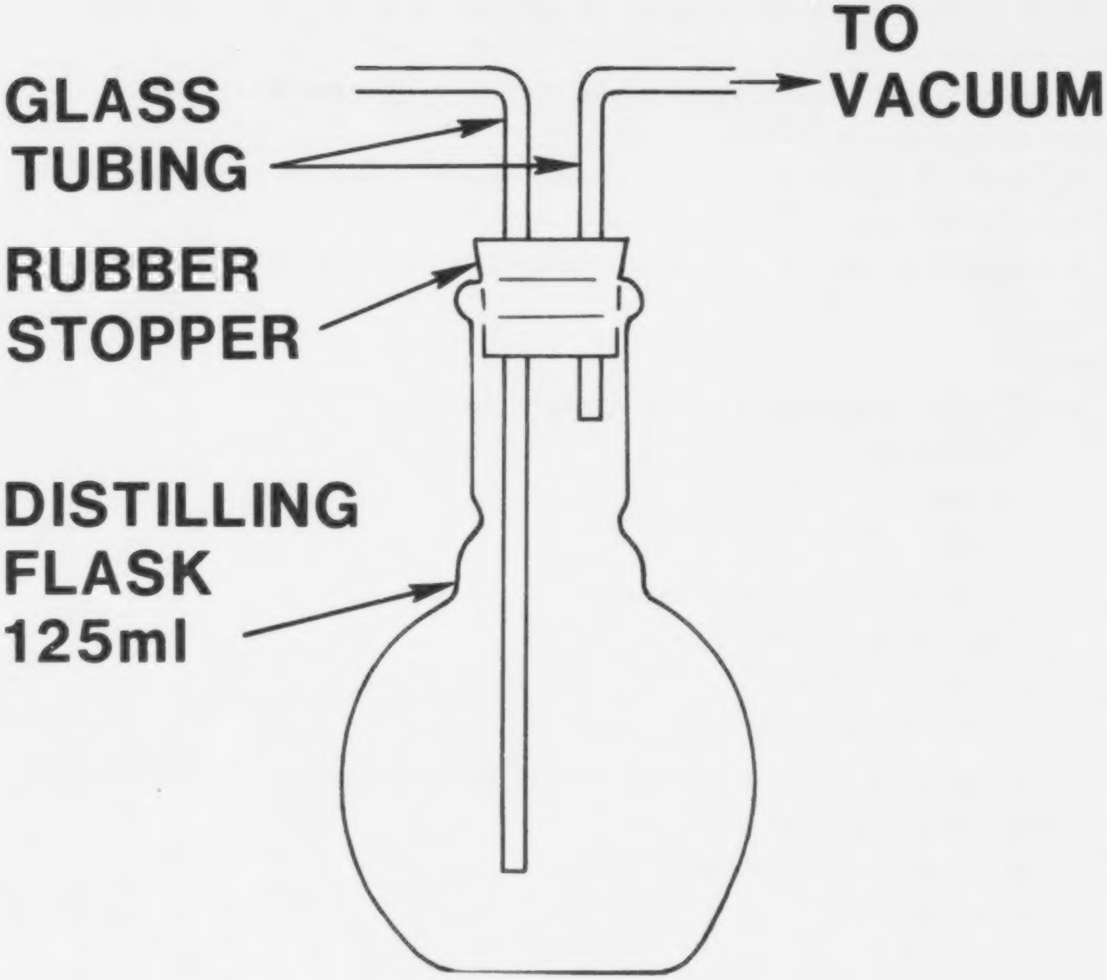
Instructions for preparing the reagents may be found in section I of the Training Manual outline, Laboratory Determination of Oil and Grease.

5 ml 50% hydrochloric or sulfuric acid

1 g anhydrous sodium sulfate (See IX B, second item)

95 ml organic extracting solvent. Acceptable grades are:

Dupont's Freon TF, Allied's Genosolv D, and Allied's Genetron 113.


TO VACUUM
GLASS TUBING
RUBBER STOPPER
DISTILLING FLASK 125ml

NAME ____________________________

QUIZ

Oil and Grease

Check the correct answer or fill in the missing word(s) as indicated.

1. The definition of oil/grease is clear.

   ____ T

   ____ F

2. Three classes of compounds generally referred to as oil or grease are, __________________, ______________, and ___________________.

3. Two examples of industries which may produce oil/grease wastes are ___________________ and ____________________.

4. Two problems caused by oil/grease in wastewater treatment plants are ___________________ and ____________________.

5. Any organic extracting solvent may be used in determining oil/grease.

   ____ T

   ____ F

6. In the NPDES procedure, the extracting solvent is

   ____ Chloroform

   ____ Hexane

   ____ Freon

7. The extraction is performed using a

   ____ Kuderna-Danish apparatus

   ____ steam distillation

   ____ a separatory funnel

8. Given the following information, what is the value for oil/grease in mg/l?

   wgt of flask = 52.1620 g

   wgt of flask plus oil/grease = 52.1635 g

   ml of sample = 500

   ____ 300 mg/l

   ____ 30.0 mg/l

   ____ 3 mg/l

NAME ____________________

QUIZ - ANSWER SHEET

Oil and Grease

Check the correct answer or fill in the missing word(s) as indicated.

1. The definition of oil/grease is clear.

   ____ T

   __X__ F

2. Three classes of compounds generally referred to as oil or grease are, __waxes__, __fatty acids__, and __fats; or oil, hydrocharbons, animal and vegetable glycerides__.

3. Two examples of industries which may produce oil/grease wastes are __meat processers__, and __petroleum refineries; or petro chemicals, trucking, laundry, machine tool, steel__.

4. Two problems caused by oil/grease in wastewater treatment plants are __safety__, and __odor; or inhibit biodegradation__.

5. An organic extracting solvent may be used in determining oil/grease.

   ____ T

   __X__ F

6. In the NPDES procedure, the extracting solvent is

   _____ Chloroform

   _____ Hexane

   __X__ Freon

7. The extraction is performed using a

   ____ Kuderna-Danish apparatus

   ____ steam distillation

   ____ a separatory funnel

8. Given the following information, what is the value for oil/grease in mg/l?

   ____ 300 mg/l

   ____ 30.0 mg/l

   __X__ 3 mg/l

Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For"fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

Give partial credit for use of correct formula, setting the problem up correctly, etc.

US EPA (LOGO)
PRESENTS

SLIDE 1

PRINTING:

THE DETERMINATION OF GREASE AND OIL, US ENVIRONMENTAL PROTECTION AGENCY, OFFICE OF WATER PROGRAMS OPERATIONS NATIONAL TRAINING AND OPERATIONAL TECHNOLOGY CENTER

SLIDE 2

PRINTING:

- PROBLEMS CAUSED BY GREASE AND OIL
- ANALYTICAL METHOD
- CALCULATIONS

SLIDE 3

PRINTING:

- ACIDIFICATION
- EXTRACTION
- SEPARATION
- EVAPORATION
- WEIGHING

SLIDE 4

DRAWING DEPICTING SCIENTIST LOOKING FOR DEFINITION OF OIL AND GREASE IN THE DICTIONARY

SLIDE 5

PRINTING:

- SAMPLE SOURCE
- SOLVENT USED
- OTHER EXTRACTABLES

SLIDE 6

PRINTING:

- HYDROCARBONS
- ESTERS
- OILS
- FATS
- WAXES
- FATTY ACIDS

SLIDE 7

PRINTING:

- GASOLINE
- FUEL OILS
- LUBRICATING OILS
- ASPHALTS

SLIDE 8

CARTOON DEPICTING OIL AND GREASE CAUSING PROBLEMS FOR WASTEWATER TREATMENT PLANT OPERATOR

SLIDE 9

DRAWING DEPICTING WASTEWATER TREATMENT PLANT OPERATOR LOOKING AT OIL/GREASE LAYER ON WATER IN TANK.

SLIDE 10

SAME AS 10 EXCEPT LAYER HAS FOUL ODOR.

SLIDE 11

CARTOON DEPICTING OXYGEN AND MICROORGANISMS UNABLE TO BREAK DOWN WASTE MATERIAL BECAUSE OF GREASE COATING.

SLIDE 12

PRINTING:

- PIPELINES
- PUMPS
- SCREENS
- FILTERS

SLIDE 13

SAME AS 10 EXCEPT OIL/GREASE IS CAUSING UNSAFE FOOTING.

SLIDE 14

PHOTO OF ANALYST WITH GLASS STOPPERED SAMPLE BOTTLE.

SLIDE 15

PHOTO OF ANALYST MARKING GLASS STOPPERED SAMPLE BOTTLE.

SLIDE 16

PRINTING:

- 5 ml OF 50% H_2SO_4
- 4°C

SLIDE 17

CARTOON DEPICTING BACTERIA TRYING TO BREAK DOWN WASTE MATERIAL.

SLIDE 18

SERIES XT-56, DETERMINATION OF OIL AND GREASE

PHOTO OF ANALYST POURING SAMPLE FROM BOTTLE INTO SEPARATORY FUNNEL.

SLIDE 19

PRINTING:

1000 ml

SLIDE 20

PRINTING:

pH2

SLIDE 21

PHOTO OF ANALYST ADDING FREON TO SAMPLE BOTTLE

SLIDE 22

PHOTO OF WATER/FREON LAYER IN SEPARATORY FUNNEL

SLIDE 23

PHOTO OF ANALYST PLACING STOPPER IN SEPARATORY FUNNEL

SLIDE 24

PHOTO OF ANALYST SHAKING SEPARATORY FUNNEL

SLIDE 25

PHOTO OF ANALYST OPENING STOPCOCK OF SEPARATORY FUNNEL

SLIDE 26

PRINTING:

1. GENTLE SHAKING
2. PRESSURE RELEASE

SLIDE 27

PRINTING:

2 MINUTES

SLIDE 28

PHOTO OF FREON-WATER INTERFACE IN SEPARATORY FUNNEL

SLIDE 29

SLIDE

SLIDE

PHOTO OF ANALYST DRAINING FREON INTO 125 ml DISTILLING FLASK.

SLIDE 30

SAME AS 30 EXCEPT FREON IS BEING DRAINED THROUGH FILTER PAPER.

SLIDE 31

PHOTO OF ANALYST ADDING MORE FREON TO THE SEPARATORY FUNNEL.

SLIDE 32

PRINTING:

1. INVERT THE FUNNEL
2. PRESSURE RELIEF
3. 5 SECOND GENTLE SHAKE
4. PRESSURE RELIEF

SLIDE 33

PRINTING:

5. REPEAT 3 AND 4
6. 2 MINUTE VIGOROUS SHAKE
7. PRESSURE RELIEF
8. LAYER SEPARATION

SLIDE 34

SAME AS 32

SLIDE 35

SAME AS 33

SLIDE 36

PRINTING:

- TIP OF FUNNEL
- FILTER PAPER

SLIDE 37

PRINTING:

- WATER BATH
- 70°C
- 80°C

SLIDE 38

DRAWING OF 125 ml DISTILLING FLASK WITH DEVICE FOR APPLYING VACUUM.

SLIDE 39

PHOTO OF ANALYST DISTILLING FREON FROM THE 125 ml DISTILLING FLASK.

SLIDE 40

PHOTO OF ANALYST PLACING 125 ml FLASK INTO DESICCATOR.

SLIDE 41

PRINTING:

mg OIL OR GREASE/l =

$$\frac{(A - B) \times 1000 \times 1000}{C}$$

SLIDE 42

PRINTING:

mg OIL OR GREASE/l =

$$\frac{(A - B) \times 1000 \times 1000}{C}$$

A = WT. OF FLASK + RESIDUE EXPRESSED IN G

SLIDE 43

PRINTING:

mg OIL OR GREASE/l =

$$\frac{(A - B) \times 1000 \times 1000}{C}$$

B = WEIGHT OF FLASK EXPRESSED IN G

SLIDE 44

PRINTING:

mg OIL OR GREASE/l

$$\frac{(A - B) \times 1000 \times 1000}{C}$$

1000 CONVERTS TO ml TO l

SLIDE 45

PRINTING:

mg OIL OR GREASE/l =

$$\frac{(A - B) \times 1000 \times 1000}{C}$$

1000 CONVERTS g TO mg

SLIDE 46

PRINTING:

mg OIL OR GREASE/l =

$$\frac{(A - B) \times 1000 \times 1000}{C}$$

C = ml OF SAMPLE

SLIDE 47

PRINTING:

- PROBLEMS CAUSED BY GREASE AND OIL
- ANALYTICAL METHOD
- CALCULATIONS

SLIDE 48

PRINTING:

AUTHOR: C. R. FELDMANN

TECHNICAL REVIEW: P. F. HALLBACH
R. C. KRONER

NARRATION: C. R. FELDMANN

GRAPHICS: GRAPHICS ARTS
NERC, CINCINNATI

SLIDE 49

CLEAN
WATER

SLIDE 50 (LAST SLIDE)

SLIDE

SLIDE

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Phenol

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 2 hours, 25 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant should have completed one year of college organic chemistry, or equivalent, and have the ability to:

A. Handle solutions of acid safely.

B. Understand the terms µg, mg, g, ml, and l as metric terms of mass and volume.

C. Perform simple calculations (addition, subtraction, multiplication, and division) involving decimals.

D. Use a Spectronic 20 (or equivalent) spectrophotometer.

E. Prepare a calibration graph and use it to determine the concentration of a sample constituent.

F. Perform a distillation.

G. Use ordinary laboratory glassware (beakers, flasks, graduated cylinders, graduated pipets, volumetric pipets, etc.).

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on the Subject Matter, will obtain absorbance values for the assigned phenol standards, use them with the corresponding concentration values to prepare a calibration graph, and then use the graph to determine the phenol concentration of the sample.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual, and the equipment/supplies/reagents listed in sections IX and X of this IPW.

C. Accepted Performance:

1. A grade of 70% on the quiz.

2. The instructor will judge whether or not the calibration graph has been prepared properly (e.g., use of the entire x and y axes,

and line of best fit).

3. Value for phenol concentration of sample within ± 10% of class average.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Two outlines in the Training Manual:
    - a. Determination of Phenolics
    - b. Laboratory Determination of Phenol-Direct Photometric Method (according to Standard Methods for the Examiniation of Water and Wastewater, 14th edition, page 582, 1975)
2. X-37, Phenols (4 slides); See XI, Description of Visual Materials (page 25-8).
3. XT-28, Determination of Phenolics (65 slides, 17 minute tape, script); See XI, Description of Visual Materials (page 25-9).

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
2. Review the laboratory procedure by performing the student assignment.
3. Reproduce the attached quiz; one copy for each participant, plus any desired file copies. See page 25-6 and 25-7.
4. Prepare the equipment/supplies/reagents listed in Sections IX and X of this IPW.
5. Obtain a sample of municipal wastewater treatment plant effluent. A maximum of 500 ml per laboratory station will be needed.
6. Each laboratory station will run the 0.0, 2 other standards, and the sample. Determine which stations will run which other standards. A "composite" calibration graph will be prepared.
7. Prepare a chart on the chalkboard for the data.

B. Sequencing:

1. Classroom - discuss phenol using X-37, Phenols.
    - a. Two dimensional formulas for benzene, the compound from which phenol may be derived; X-37, 1.
    - b. Formulas for phenolic compounds; X-37, 2.
    - c. Reaction of phenol with 4 - AAP; X-37, 3.
    - d. Locating substituents on the benzene ring; X-37, 4.
2. Classroom - introduce and present XT-28, Determination of Phenolics.
    - a. Introduction; XT-28, 1-3.
    - b. Formulas for phenolics; XT-28, 4-5.

   c. Importance of phenolics; XT-28, 6.
   d. Sources and uses of phenolics; XT-28, 7-12.
   e. Problems caused by the presence of phenolics in water; XT-28, 13-17.
   f. Compounds produced by the chlorination of phenol; XT-28, 18-22.
   g. Summary of the determination; XT-28, 23.
   h. Adjustment of sample pH and removal of interferences; XT-28, 24-26.
   i. Sample collection and preservation; XT-28, 27-33.
   j. Sample distillation; XT-28, 34-35.
   k. Colorimetric determination; XT-28, 36-45.
   l. Analytical response of various types of phenolics; XT-28, 46-48.
   m. Other interferences in the determination; XT-28, 49-57.
   n. Summary and credits; XT-28, 58-65.

3. Classroom - note analytical procedures other than for NPDES; Section IV B of the outline, Determination of Phenolics.
4. Classroom - note test variables; Section VI C, same outline.
5. Classroom - discuss accuracy and precision of the test; Section VIII, same outline.
6. Classroom - make the assignments determined in VIII A. 5. above.
7. Laboratory - have the participants do Section II of the Training Manual outline, Laboratory Determination of Phenol - Direct Photometric Method. Paragraph F of Section II calls for distilling 450 ml of sample (in accordance with Standard Methods). Since the distillation is rather time consuming, tell the participants to distill only 250 ml, and to delete paragraphs I and J. Emphasize strongly that this "short-cut" is being taken for agenda time considerations only, and that for actual samples, the "short-cut" would lead to low results.
8. Classroom - determine which absorbance values will be used by the class to prepare the calibration graph.
9. Classroom - have the participants do Section III of the same outline (B. 7. above).
10. Classroom - final discussion and questions.
11. Classroom - give the quiz and then read correct answers from the attached answer sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 1 sample, the 0.0 standard, and 2 other standards.

A. For Each Station:

1 pair safety glasses (for each person at the station)

1 laboratory apron or coat (for each person at the station)

1 distilled water plastic squeeze bottle

1 grease pencil (or other marking pencil)

paper towels or sponge

1 distilling apparatus, consisting of:

- 1 ring stand
- 1 ring (to support a 1 liter round bottom flask)
- 2 3-finger clamps
- 1 1 liter Erlenmeyer flask
- 1 wire gauze
- 1 Fisher burner (or equivalent)
- 1 1 liter round bottom flask with 24/40 joint and long neck; SGA JF-2130 or equivalent and containing 6-10 glass beads.
- 1 offset adapter with 24/40 joints and small stopper to plug the open hole; SGA JA-4950 or equivalent.
- 1 40 cm condenser with tubing, SGA JC-9375 or equivalent

1 500 ml graduated cylinder

1 1 liter beaker

1 5.0 ml volumetric pipet

1 small funnel (to fit the hole in the distillation apparatus adapter)

4 100 ml volumetric flasks

Volumetric or graduated pipets; to prepare the assigned standards

1 pipet bulb

1 50 ml graduated cylinder

4 125 ml Erlenmeyer flasks

2 2 ml volumetric pipets

B. Shared:

Spectronic 20 (or equivalent) spectrophotometer and 1 cm cell; 1 per 4 stations with a large beaker for rinsing the cell.

pH meter with pH 9, 10, or 11 buffer and a small beaker to hold the electrode(s)

Chalkboard, chalk, and erasers for posting data

X. IPW REAGENT REQUIREMENTS:

Minimum Amounts for Each Station
(Prepare excess reagent to allow for waste, rinsing of glassware, and any desired re-runs.)

Instructions for preparing the reagents may be found in section I of the Training Manual outline, Laboratory Determination of Phenol - Direct Photometric Method.

1 ml methyl orange*

1 ml phosphoric acid*

5 ml copper sulfate

Intermediate phenol standard; volumes as assigned

8 ml ammonium chloride

8 ml potassium ferricyanide

8 ml 4-aminoantipyrine

100 ml pH 9, 10, or 11 buffer

*In eyedropper bottles

NAME______________________________

QUIZ
Phenol

Check the correct answer or fill in the missing word(s) as indicated.

1. Phenolic compounds are defined as hydroxy derivatives of benzene.

   ______T

   ______F

2. Even in small concentrations, phenolic compounds can impart an unpleasant ____________________and____________________ to fish and drinking water.

3. The color developing reagent in the NPDES method for determining phenolics is ______________________________.

4. In the NPDES method for determining phenolics, ________________ is commonly used to remove interferences.

5. The pH is unimportant in the colorimetric NPDES procedure for determining phenolics.

   ______T

   ______F

6. Even though many derivatives of phenol exist, which compound is used as the standard in the NPDES method for determining phenolics?

   ____

NAME________________________

QUIZ - ANSWER SHEET
Phenol

Check the correct answer or fill in the missing word(s) as indicated.

1. Phenolic compounds are defined as hydroxy derivatives of benzene.

   __X__ T

   _____ F

2. Even in small concentrations, phenolic compounds can impart an unpleasant __taste__ and __odor__ to fish and drinking water.

3. The color developing reagent in the NPDES method for determining phenolics is __4-aminoantipyrine, or 4-AAP__.

4. In the NPDES method for determining phenolics, __distillation__ is commonly used to remove interferences.

5. The pH is unimportant in the colorimetric NPDES procedure for determining phenolics.

   _____ T

   __X__ F

6. Even though many derivatives of phenol exist, which compound is used as the standard in the NPDES method for determining phenolics?

   Phenol________________

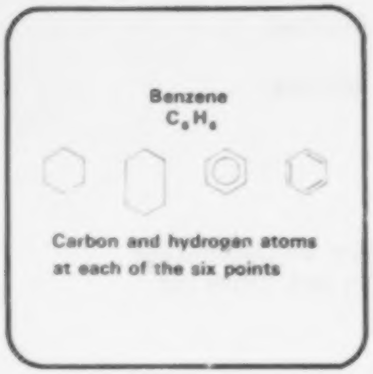
Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For "fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

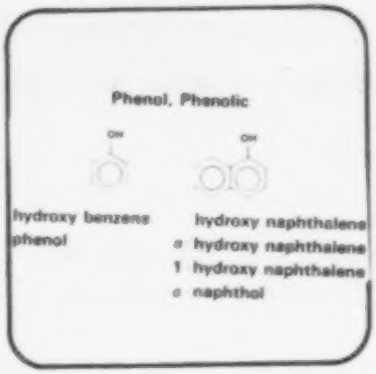
XI. DESCRIPTION OF VISUAL MATERIALS

SERIES X-37, PHENOLS


Benzene
C_6H_6

Carbon and hydrogen atoms at each of the six points

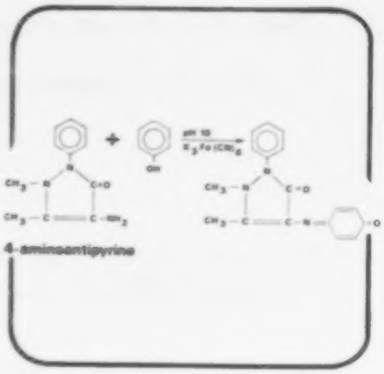
SLIDE 1


Phenol, Phenolic

hydroxy benzene
phenol

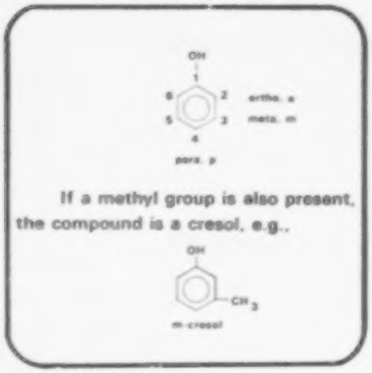
hydroxy naphthalene
α hydroxy naphthalene
1 hydroxy naphthalene
α naphthol

SLIDE 2


+
OH
pH 10
$K_3Fe(CN)_6$
N
CH_3 — N
C=O
CH_3 — C
C — NH_2
N
CH_3 — N
C=O
CH_3 — C
C — N
=O

4-aminoantipyrine

SLIDE 3


OH
1
2
3
4
5
6
ortho, o
meta, m
para, p

If a methyl group is also present, the compound is a cresol, e.g.,

OH
CH_3
m-cresol

SLIDE 4 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

US EPA (LOGO)

PRESENTS

SLIDE 1

DETERMINATION OF PHENOLICS

SLIDE 2

WATER QUALITY

↑

PHENOLS

↓

ANALYTICAL PROCEDURES

SLIDE 3

DRAWING SHOWING TWO DIMENSIONAL STRUCTURAL FORMULA FOR CARBOLIC ACID

SLIDE 4

DRAWING SHOWING TWO DIMENSIONAL STRUCTURAL FORMULAS FOR EIGHT PHENOLIC COMPOUNDS

SLIDE 5

PHOTO OF

INDUSTRIAL PLANT AND RECIEVING STREAM

SLIDE 6

PRINTING:

COAL TAR

SLIDE 7

PHOTO OF

PHENOL PRODUCING PLANT

SLIDE 8

PHOTO OF

ITEMS MADE FROM PHENOL

SLIDE 9

PRINTING:

OCCURRENCE IN WASTES

NATURAL COMPONENT → IN FUEL INDUSTRIES

RAW MATERIAL → SYNTHESIS OF ORGANICS

SLIDE 10

DRAWING SHOWING TWO DIMENSIONAL STRUCTURAL FORMULAS FOR O-, M-, AND P- CRESOL

SLIDE 11

PHOTO OF

CAN OF LYSOL

SLIDE 12

PRINTING:

BIOLOGICAL TREATMENT	25 mg/l
OF	
PHENOLIC WASTES	500 mg/l

SLIDE 13

PRINTING:

EFFECT OF CHLORINE IN TREATED WATER CONTAINING TRACE QUANTITIES OF PHENOLIC COMPOUNDS

CHLORINATED PHENOLIC COMPOUNDS

SLIDE 14

PRINTING:

- AFFECT FISH
- WATER QUALITY

SLIDE 15

PHOTO OF FISHES WITH PHENOLIC POLLUTION INDICATED AS BEING PRESENT.

SLIDE 16

CARTOON DEPICTING FOUL TASTING DRINKING WATER.

SLIDE 17

PRINTING:

PROGRESSIVE CHLORINATION

OF

PHENOL

SLIDE 18

SERIES XT-28, DETERMINATION OF PHENOLICS

DRAWING SHOWING TWO DIMENSIONAL STRUCTURAL FORMULAS OF COMPOUNDS FORMED BY CHLORINATION OF PHENOL.

SLIDE 19

DRAWING OF TWO DIMENSIONAL STRUCTURAL FORMULAS SHOWING FORMATION OF 2, 6 - DICHLORPHENOL.

SLIDE 20

DRAWING OF TWO DIMENSIONAL STRUCTURAL FORMULAS SHOWING FORMATION OF 2, 4 - DICHLOROPHENOL.

SLIDE 21

DRAWING OF TWO DIMENSIONAL STRUCTURAL FORMULAS SHOWING FORMATION OF 2 - CHLOROPHENOL

SLIDE 22

PRINTING:

ANALYSIS OF PHENOLICS

- DISTILLATION
- COLORIMETRIC DETERMINATION

SLIDE 23

PRINTING:

PHOSPHORIC ACID
pH 4.0
PREVENTS
CHEMICAL OXIDATION

SLIDE 24

PRINTING:

COPPER SULFATE
1 GRAM/LITER
PREVENTS
BIOCHEMICAL OXIDATION

SLIDE 25

PRINTING:

COPPER SULFATE
1 GRAM/LITER
ELIMINATES
INTERFERENCES FROM SULFUR
COMPOUNDS

SLIDE 26

PRINTING:

ANALYSIS WITHIN

4 HOURS

SLIDE 27

PRINTING:

SAMPLE PRESERVATION

SLIDE 28

PHOTO OF pH METER USED FOR SAMPLE pH ADJUSTMENT

SLIDE 29

PHOTO OF COPPER SULFATE BEING ADDED TO SAMPLE

SLIDE 30

PHOTO OF SAMPLE BEING STORED IN REFRIGERATOR

SLIDE 31

PRINTING:

PRESERVED SAMPLES SHOULD BE ANALYZED WITHIN 24 HOURS

SLIDE 32

PRINTING:

ANALYSIS OF PRESERVED SAMPLES

- CHECK pH = 4.0
- NO ADDITIONAL COPPER SULFATE REQUIRED

SLIDE 33

PHOTO OF DISTILLATION APPARATUS

SLIDE 34

PRINTING:

- DISTILLATION RATE GRADUAL
- DISTILLATE VOLUME MUST EQUAL THAT OF SAMPLE BEING DISTILLED

SLIDE 35

PRINTING:

QUANTITATIVE ANALYSIS
OF
PHENOLIC COMPOUNDS

SLIDE 36

WORD CHEMICAL REACTION OF
PHENOLICS WITH COLORIMETRIC
REAGENTS.

SLIDE 37

PHOTO OF
STANDARD METHODS
BOOK

SLIDE 38

PRINTING:

SIMILARITIES
- REACTION CHEMICALS
- COLORIMETRIC MEASUREMENTS
- PHENOL STANDARD

SLIDE 39

PRINTING:

DIFFERENCES
- RANGE OF APPLICABILITY
- CONCENTRATING THE DYE
- WAVELENGTH
- UNIT TO EXPRESS RESULTS

SLIDE 40

PRINTING:

RANGE OF APPLICABILITY

METHOD B
LESS THAN 1.0 mg/LITER

METHOD C
MORE THAN 1.0 mg/LITER

SLIDE 41

PRINTING:

DYE CONCENTRATION

METHOD B
CONCENTRATED BY EXTRACTION WITH CHLOROFORM

METHOD C
IN ORIGINAL AQUEOUS SOLUTION

SLIDE 42

PRINTING:

WAVELENGTH

METHOD B
460 NANOMETERS (nm)

METHOD C
510 NANOMETERS (nm)

SLIDE 43

PRINTING:

CONCENTRATION UNIT

METHOD B
MICROGRAMS(μg)
PER LITER

METHOD C
MILLIGRAMS(mg)
PER LITER

SLIDE 44

PRINTING:

CALIBRATION CURVE

PHENOL STANDARD

SLIDE 45

PRINTING:

COMPOUND	DENSITY COMPARED TO PHENOL %
PHENOL	100
O-CRESOL	74
M-CRESOL	69
P-CRESOL	3

SLIDE 46

PRINTING:

PHENOLICS

UNPREDICTABLE PROPORTIONS

SLIDE 47

PRINTING:

MIXTURE OF PHENOLICS

- PHENOL AS STANDARD
- MINIMUM CONCENTRATION OF PHENOLICS

SLIDE 48

PRINTING:

INTERFERENCES

SLIDE 49

PRINTING:

INTERFERENCES

1. SULFUR COMPOUNDS
2. BACTERIA
3. ALKALINITY
4. OXIDIZERS
5. OILS AND TARS

SLIDE 50

PRINTING:

1 SULFUR COMPOUNDS

REACT WITH COPPER SULFATE IN ACID CONDITIONS→COPPER SULFIDE↓ ELIMINATES INTERFERENCES IN COLOR DETERMINATIONS

SLIDE 51

PRINTING:

1 SULFUR COMPOUNDS

- PHOSPHORIC ACID pH LESS THAN 4.0
- AERATION - STIRRING
- COPPER SULFATE

SLIDE 52

PRINTING:

2 BACTERIA

- COPPER SULFATE INHIBITS BACTERIA
- COPPER ION EXISTS IN ACID CONDITIONS

SLIDE 53

PRINTING:

3 ALKALINITY

- PHOSPHORIC ACID pH = 4.0
- PREVENTS CUPRIC HYDROXIDE, AN OXIDIZER

SLIDE 54

PRINTING:

4 OXIDIZERS

REMOVE
IMMEDIATELY
AFTER
SAMPLING

SLIDE 55

PHOTO OF BOTTLES OF FERROUS SULFATE AND SODIUM ARSENITE

SLIDE 56

PHOTO OF THREE SEPARATORY FUNNELS IN A RACK.

SLIDE 57

PRINTING:

- CHARACTERISTICS AND SOURCES

SLIDE 58

PRINTING:

- CHARACTERISTICS AND SOURCES
- EFFECT ON FISH AND WATER QUALITY

SLIDE 59

PRINTING:

- CHARACTERISTICS AND SOURCES
- EFFECT ON FISH AND WATER QUALITY
- SAMPLE PRESERVATION AND ANALYSIS

SLIDE 60

PRINTING:

- CHARACTERISTICS AND SOURCES
- EFFECT ON FISH AND WATER QUALITY
- SAMPLE PRESERVATION AND ANALYSIS
- REMOVAL OF INTERFERENCES

SLIDE 61

PRINTING:

PRECISION

- INTERFERENCES
- SKILL OF THE ANALYST
- CONCENTRATION OF PHENOLICS

SLIDE 62

PRINTING:

ACCURACY

- APPROXIMATION
- MINIMUM AMOUNT OF PHENOLICS

SLIDE 63

SERIES XT-28, DETERMINATION OF PHENOLICS

PRINTING:

TECHNICAL REVIEW

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A. E. DONAHUE R. C. KRONER

C. R. FELDMANN F. J. LUDZACK

GRAPHICS COORDINATION

B. G. KRAMIG

M. D. STREFF

NARRATION - A. E. DONAHUE

SLIDE 64

PRINTING:

CLEAN
WATER

SLIDE 65 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

SLIDE

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Polychlorinated Biphenyls

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 2 hours

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR: The participant will have completed one year of college organic chemistry, or equivalent and have successfully:

A. Understand the terms ng, µg, mg, g, ul, ml, and l as metric terms of mass and volume.

B. Met the objectives in the IPW on Introduction to Gas Chromatography.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participant will take a quiz on the Subject Matter, will determine the rentention time (RT) of p, p' - DDE, and use the RT to qualitatively determine an unknown Aroclor in a laboratory demonstration.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual and the equipment/supplies/reagents listed in sections IX and X of this IPW.

C. Accepted Performance:

1. A grade of 70% on the quiz.
2. The instructor will judge whether or not correct qualitative identification has been made.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

One outline in the Training Manual:

Method for Polychlorinated Biphenyls (PCB's) in Industrial Effluents (According to procedure by the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio)

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson, using the Sequencing below or your own organization of the material.
2. Review the laboratory procedure by performing the student assignment.
3. Reporduce the attached quiz; one copy for each participant, plus any desired file copies. See pages 26-5 and 26-6.
4. Prepare the equipment/supplies/reagents listed in Sections IX and X of this IPW.
5. Order p, p' - DDE and Aroclor. See X.2. and X.3.
6. Check the gas chromatograph for proper functioning. Inject the p, p' - DDE and Aroclor unknown so as to determine proper volumes and attenuation to be used by the participants.

B. Sequencing:

1. Classroom - discuss
   - a. What is biphenyl
   - b. 210 theoretically possible PCB's
   - c. 1970 sales of 34,000 tons; currently 400,000 tons
   - d. Uses of PCB's
   - e. Causes of concern
   - f. Method of analysis
2. Laboratory -
   - a. Have the participants inject the p, p' - DDE standard and determine its RT from the first appearance of the solvent peak. Tell the participants what volume to inject.
   - b. Have the participants inject the unknown Aroclor twice, and time the appearance of each peak. Tell the participants what volume to inject.
   - c. Have the participants determine the RT for each peak, using the average of the two times.
   - d. Have the participants use the RT to qualitatively determine the identity of the Aroclor.
3. Classroom - final discussion and questions.
4. Classroom - give the quiz and then read the correct answers from the attached answer sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 1 sample

A. For Each Station:

1 pair safety glasses (for each person at the station)

1 laboratory apron or coat (for each person at the station)

B. Shared:

1. Gas chromatograph; 1 per 2 stations with:
    a. electron capture detector (nickel 63 or tritium)
    b. column
        1) glass (180 cm long by 4 mm internal diameter)
        2) solid support - gas chrom Q (100-200 mesh)
        3) liquid phase - 3% SE 30, or 3% OV 1, or 1.5% OV 17 + 1.95% QF 1
    c. rotometers - for monitoring carrier gas flow
    d. recorder - potentiometric strip chart (10 in.); compatible with detector
    e. chart paper for recorder
2. Carrier gas; one each of a, b, and c per 2 gas chromatographs:
    a. argon-methane (95%-5%) for pulsed detector, or nitrogen (purified grade) for direct current.
    b. pressure regulator; two stage CGA 580 for nitrogen, or two stage CGA 350 for argon-methane
    c. filter for carrier gas; with
        1) oxygen trap
        2) water trap
3. Micro syringe (10 µl); 1 per gas chromatograph.
4. Stopwatch (or equivalent); 1 per gas chromatograph.
5. Tissues; several per gas chromatograph.
6. Ruler (12 inches); 1 per gas chromatograph.

X. IPW REAGENT REQUIREMENTS:

Minimum Amounts for Each Station
(Prepare excess reagent to allow for waste, rinsing of glassware, and any desired re-runs.)

1. Hexane (pesticide quality) for rinsing syringes; about 10 ml in a small glass stoppered bottle at each gas chromatograph.

2. p, p' - DDE pesticide standard*(in hexane), about 0.1 ng/μl; about 5 ml in a small glass stoppered bottle at each gas chromatograph.

3. Aroclor* (in hexane), about 0.1 ng/μl; about 5 ml in a small glass stoppered bottle at each gas chromatograph.

*Aroclor and DDE standards may be obtained by writing to: USEPA, EMSL, Quality Assurance Branch, Cincinnati, Ohio 45268.

NAME______________________________

QUIZ

Polychlorinated Biphenyls

Check the correct answer or fill in the missing word(s) as indicated.

1. PCB's may be considered as chlorine derivatives of

   __________methane

   __________toluene

   __________phenol

   __________biphenyl

2. How may different types of PCB's are theoretically possible?

   _____28

   _____2695

   _____210

   _____5

3. Two reasons for being concerned about the presence of PCB's in the environment are their ____________________ and ____________________.

4. PCB's are widly used because of their flame ____________________properties.

5. Use of a ____________________ column is one method of "cleaning up" the sample in a PCB determination.

NAME____________________________

QUIZ - ANSWER SHEET
Polychlorinated Biphenyls

Check the correct answer or fill in the missing word(s) as indicated.

1. PCB's may be considered as chlorine derivatives of

__________methane
__________toluene
__________phenol
____X____biphenyl

2. How may different types of PCB's are theoretically possible?

_____28
_____2695
__X__210
_____5

3. Two reasons for being concerned about the presence of PCB's in the environment are their persistance and toxicity.

4. PCB's are widly used because of their flame retardant properties.

5. Use of a florisil or silica gel column is one method of "cleaning up" the sample in a PCB determination.

Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For "fill-in" answers, any which are equivalent to those indicated above would, of course, also be correct.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Control of Analytical Performance

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 30 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: An analyst must know how to control or quanitiate errors in laboratory results and how to check for precision (reproducibility) and accuracy (recovery of a constituent) to substantiate the reliability and validity of the data. He/she must know and practice requirements for quality assurance for every phase of laboratory work so the data will be precise and accurate.

V. ENTRY LEVEL BEHAVIOR: The participant should have the ability to use formulas and perform basic calculations, fundamental knowledge of analytical chemistry and equipment and supplies normally found in a chemistry laboratory, at least six months experience in chemical laboratory work and completion of the lessons on sample handling and on compliance methodology. The lesson will be most effective if participants have read the outlines on the topics prior to the lesson.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior:

The participants will know the difference between accuracy and precision; the causes, effects and means to detect/quantitate sources of errors; the names, significance and some of the formulas for related statistics; where to find procedures to validate data for accuracy and precision; how to utilize a provided outline of items to be controlled to assure quality in laboratory work. Also, the participant will take a quiz on the Subject Matter.

B. Conditions:

He/she will have heard the lecture on the Subject Matter, and will have the use of the Training Manual.

C. Accepted Performance:

A grade of 70% on the quiz.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the Training Manual: Statistics for Chemists, Accuracy-Precision-Error, and Elements of a Quality Assurance Program.

2. X-24, Precision (20 slides); See XI, Description of Visual Materials (pages 27-9 through 27-11).
3. X-25, Accuracy (14 slides); See XI, Description of Visual Materials (pages 27-12 and 27-13).

4. X-26, Quality Assurance (5 slides); See XI, Description of Visual Materials (page 27-14).

5. Three handouts - Copies are at the end of this IPW:

   a. Two pages on the "Method of Standard Additions" (pages 25-15 & 25-16).

   b. One page on "Example of Calculating Percent Recovery for a Spike" (page 27-17).

   c. One page with examples of significant figures and basics on "rounding off" and calculating rules (page 27-18).

6. Two handouts available from US EPA Regional Quality Assurance Coordinator or US EPA-EMSL (See 7.).

   a. List of US EPA's currently available, free, quality control samples.

   b. List of US EPA's Regional Quality Assurance Coordinators (Optional - if students are from different parts of the country. Otherwise announce information about their regional contact).

7. Resource, US EPA, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EMSL, Cincinnati, Ohio 45268. Copies can be ordered from EMSL: 513/684-7301.

B. Suggested Media:

None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the Available Media and prepare the lesson using the Sequencing below or your own organization of the material.

2. Duplicate handout materials; one copy of each item for each participant, plus any desired file copies.

3. Reproduce the attached quiz; one copy for each participant plus any desired file copies.(See pages 27-7 and 27-8)

B. Sequencing:

Classroom - discuss outline in Training Manual, Elements of a Quality Assurance Program.

1. Introduction - 10 minutes

Highlights and cautions on:

a. Outline Section IC, Elements (to give overview)

b. Outline Section II, Sample (Just list the points since completion of the lesson on Sample Handling is a prerequisite for this lesson).

c. Outline Section III, Recognized Methodology (simply note, since discussion of this is a prerequisite for this lesson)

d. Outline Section IV, Control of Services, Instruments, Equipment and Supplies (Highlights and Cautions).

2. Outline Section V, Quality Analytical Performance: Precision - 35 minutes; using selected slides from X-24, Precision, and X-26, Quality Assurance.

   a. Analytical Results are imperfect; X-24, 1. (The Accuracy-Precision-Error outline discusses the material related to slides 1-4).

   b. Define/exemplify accuracy. Results are close to the true value-standard. Accuracy can also be checked by recovery of a constituent - spike); X-24, 2.

   c. Define/exemplify precision, and contrast it to accuracy; X-24, 3. Stress that both accuracy and precision are requirements for reliable data.

   d. Classification and causes of error; X-24, 4.

   e. Normal distribution; X-24, 5. (The Statistics for Chemists outline has reference information for slides 5-16, 18, 19).

   f. Normal distribution is a population characteristic. An analyst deals with a sample of a population. Statistics are derived from samples; X-24, 6.

   g. Define statistic; X-24, 7.

   h. Definition of mean; X-24, 8.

   i. Formulas for variance and standard deviation; X-24, 9.

   j. Calculating variance; X-24, 10-12.

   k. Calculating standard deviation from variance; X-24, 13.

   l. Calculating standard deviation on a calculator; x-24, 14.

   m. Relationship between standard deviation and normal distribution; X-24, 15.

n. Checking for normal distribution - using probability paper; X-24, 16.

o. Outline Section V B. Establishing Analyst Precision: X-24, 17.

p. $\bar{X}$ and s control chart for precision based on data from one check standard. Plot succeeding results; X-26, 3.

q. Formula for coefficient of variation (% Relative standard deviation); X-24, 18.

r. Definition of range; X-24, 19.

s. Range can be used to estimate standard deviation. "d_n" - depends on number of data items; X-24, 20.

t. $\bar{R}$ & R estimates of limits on control chart for precision based on data from dupicates. Plot succeeding ranges of duplicates; X-26, 2.

u. Summary remarks on precision - 5 minutes.

Recommend daily check by running a duplicate every 10 samples.

3. Short break may be inserted here.

4. Outline Section V, Quality Analytical Performance: Accuracy - 25 minutes; using selected slides from X-25, Accuracy, and X-26, Quality Assurance.

a. Effects of determinate error; X-25, 5. (Reference information for slides 5 and 6 are in the Accuracy-Precision-Error outline).

b. Detection of determinate error with unknowns; X-25, 6.

c. Correction of determinate error with laboratory methods; X-25, 7.

1) Hand out 2 pp. on "Method of Standard Additions" and discuss at point 3 on slide 7.

d. Percent Recovery Statistic for Accuracy:

1) Formula for using a standard to check percent recovery. The "observed" can be the average of several results; X-25, 8.

2) Formula for using a spiked sample to check percent recovery; X-25, 9.

3) Explanation of term, "background" in slide 9 formula is on X-25, 10.

a) Hand out and go over "Example of Calculating Percent Recovery of a Spike" to demonstrate correct use of the information on X-25, 9 and 10.

4) Outline Section VC. Establishing Analyst Accuracy; X-25, 11. Note that one can use a standard instead. The latter calculations are easier.

5) $\bar{X}$ and s of % recovery control chart for accuracy based on data from spiked samples. Plot succeeding % recoveries of spikes; X-26, 4.

e. Percent Bias (Percent Relative Error) Statistics for Accuracy

1) Two major reference manuals use different names for this statistic; X-25, 12.

2) Formula for using a standard to check percent bias; X-25, 13.

3) $\bar{X}$ and s of % bias control chart for accuracy based on data from spiked samples. Plot succeeding % bias (% relative error) of spikes; X-26, 5. You can also plot % bias of standards.

f. Relationship of Percent Recovery and Percent Bias Statistics; X-25, 14.

g. Summary remarks on accuracy.

Recommend daily check by running a standard or spike every 10 samples.

5. Highlights of remaining outline sections - 10 minutes.

a. Outline Section VF. Interlaboratory Checks on Performance. Encourage all 5 activities. Hand out current list of US EPA quality control samples (VII A 6. a.)

b. Outline Section VI A, The Analytical Value - give overview. For VI.A.4., hand out and discuss the sheet on significant figures. Note reference for details.

c. Outline Section VII, Safety Considerations. Encourage having a safety officer.

d. Outline Section VIII, US EPA - Analytical Quality Control Coordinators. Encourage contact; hand out current roster (VII A 6. b.) if participants are from several regions. Otherwise give them the information about their regional contact.

6. Final discussion and questions.

7. Give the quiz and then read the correct answers from the attached sheet.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

For Each Student

Handout materials

X. IPW REAGENT REQUIREMENTS:

None

NAME ______________________

QUIZ

Control of Analytical Performance

Check the correct answer or fill in the definitions as indicated.

1. Give a brief definition of precision.

2. Give a brief definition of accuracy.

3. Given the following analytical results for a standard of 10.6 mg/l:

10.2 mg/l
10.4 mg/l
10.0 mg/l
10.2 mg/l
10.7 mg/l,

a. What is the mean?

____ 10.3
____ 10.2
____ 10.4
____ 10.7
____ 10.5

b. What is the range?

____ 0.2
____ 0.3
____ 0.4
____ 0.5
____ 0.6
____ 0.7
____ 0.8

c. What is the standard deviation (estimated from range when $d_n = 2.33$)?

____ 0.3
____ 0.4
____ 0.5
____ 0.6
____ 0.7

d. What is the percent recovery?

____ 95.3%
____ 97.2%
____ 102.9%
____ 105.1%

NAME ______________________

QUIZ - ANSWER SHEET

Control of Analytical Performance

Check the correct answer or fill in the definitions as indicated.

1. Give a brief definition of precision.

   Results agree with each other.
   Ability to reproduce a result.

2. Give a brief definition of accuracy.

   Results are close to the true value.
   Agreement of a result with the true value.
   Ability to recover a known amount of spike (constituent).

3. Given the following analytical results for a standard of 10.6 mg/l:

   10.2 mg/l
   10.4 mg/l
   10.0 mg/l
   10.2 mg/l
   10.7 mg/l

   a. What is the mean?

   X 10.3
   ____ 10.2
   ____ 10.4
   ____ 10.7
   ____ 10.5

   b. What is the range?

   ____ 0.2
   ____ 0.3
   ____ 0.4
   ____ 0.5
   ____ 0.6
   X 0.7
   ____ 0.8

   c. What is the standard deviation (estimated from range when $d_n = 2.33$?

   X 0.3
   ____ 0.4
   ____ 0.5
   ____ 0.6
   ____ 0.7

   d. What is the percent recovery?

   ____ 95.3%
   X 97.2%
   ____ 102.9%
   ____ 105.1%

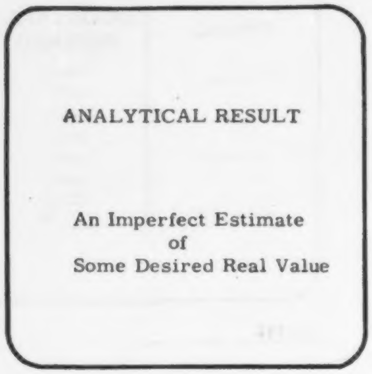
Note to Instructor:

It is suggested that one point be awarded for each correct answer.

For fill-in answers, any which are equivalent to those indicated above would, of course, also be correct.

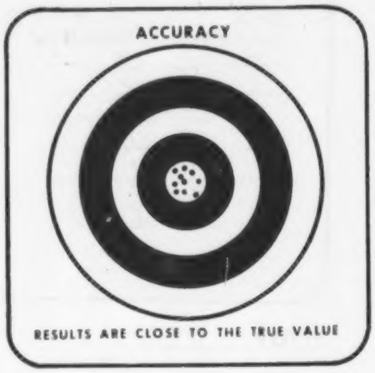
XI. DESCRIPTION OF VISUAL MATERIALS

SERIES X-24: PRECISION


ANALYTICAL RESULT

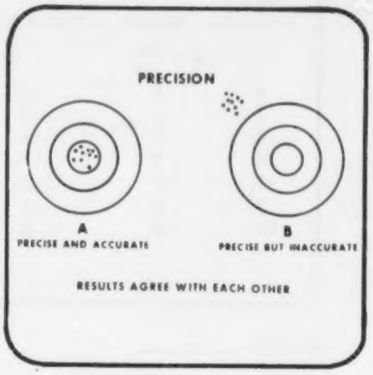
An Imperfect Estimate
of
Some Desired Real Value

SLIDE 1


ACCURACY

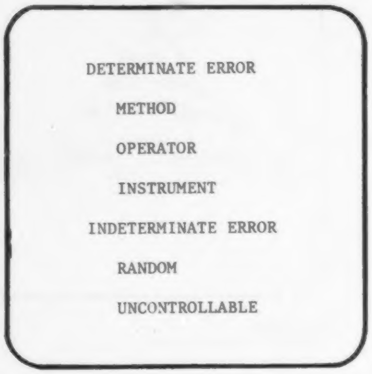
RESULTS ARE CLOSE TO THE TRUE VALUE

SLIDE 2


PRECISION

RESULTS AGREE WITH EACH OTHER

SLIDE 3


DETERMINATE ERROR

METHOD

OPERATOR

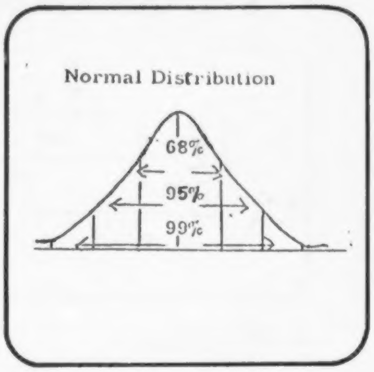
INSTRUMENT

INDETERMINATE ERROR

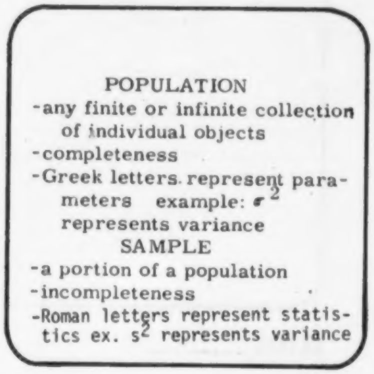
RANDOM

UNCONTROLLABLE

SLIDE 4


Normal Distribution

SLIDE 5

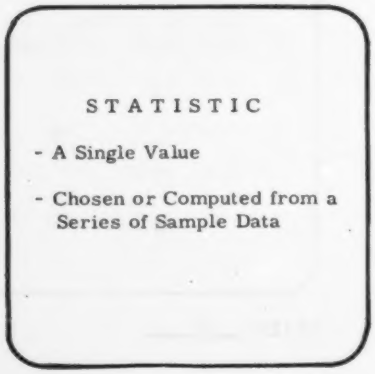

POPULATION

-any finite or infinite collection of individual objects
-completeness
-Greek letters represent parameters example: σ^2 represents variance

SAMPLE

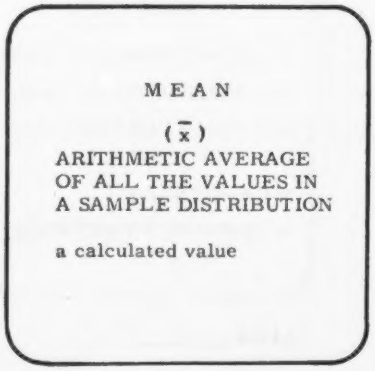
-a portion of a population
-incompleteness
-Roman letters represent statistics ex. s^2 represents variance

SLIDE 6


STATISTIC

- A Single Value
- Chosen or Computed from a Series of Sample Data

SLIDE 7

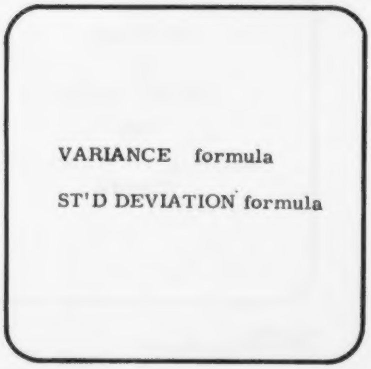

MEAN

$(\bar{x})$

ARITHMETIC AVERAGE
OF ALL THE VALUES IN
A SAMPLE DISTRIBUTION

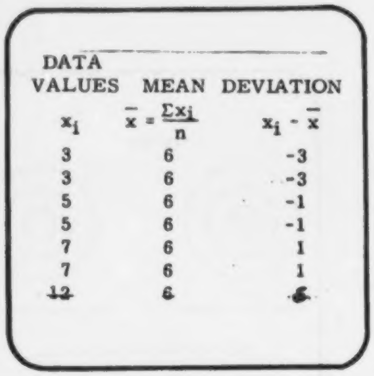
a calculated value

SLIDE 8


VARIANCE formula

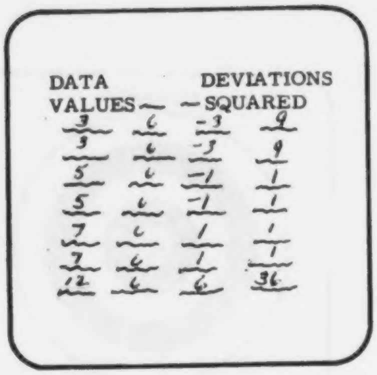
ST'D DEVIATION formula

SLIDE 9



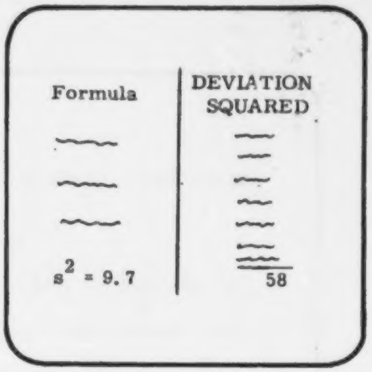
DATA VALUES x_i	MEAN $\bar{x} = \frac{\Sigma x_i}{n}$	DEVIATION $x_i - \bar{x}$
3	6	-3
3	6	-3
5	6	-1
5	6	-1
7	6	1
7	6	1
12	6	6

SLIDE 10



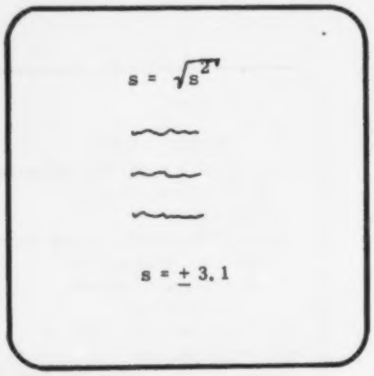
DATA VALUES			DEVIATIONS SQUARED
3	6	-3	9
3	6	-3	9
5	6	-1	1
5	6	-1	1
7	6	1	1
7	6	1	1
12	6	6	36

SLIDE 11



Formula	DEVIATION SQUARED
$s^2 = 9.7$	58

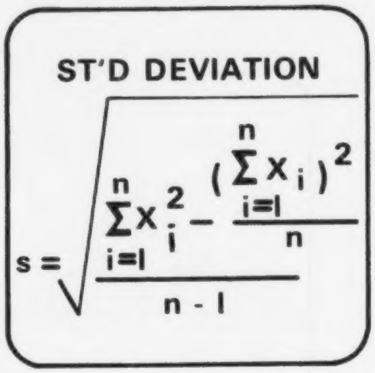
SLIDE 12



$s = \sqrt{s^2}$

$s = \pm 3.1$

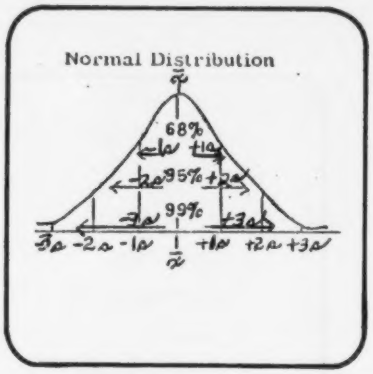
SLIDE 13



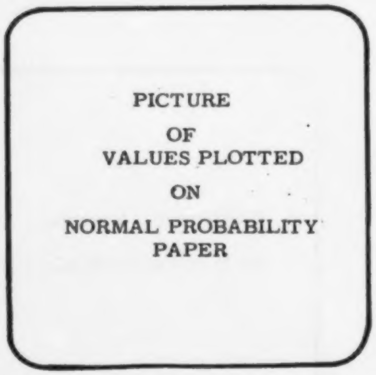
ST'D DEVIATION

$$s = \sqrt{\frac{\sum_{i=1}^{n} x_i^2 - \frac{\left(\sum_{i=1}^{n} x_i\right)^2}{n}}{n-1}}$$

SLIDE 14

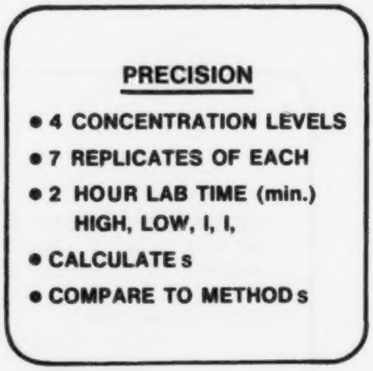

Normal Distribution
$\bar{x}$
68%
-1σ
$+1\sigma$
-2σ
95%
$+2\sigma$
99%
-3σ
$+3\sigma$
-3σ
-2σ
-1σ
$+1\sigma$
$+2\sigma$
$+3\sigma$
$\bar{x}$

SLIDE 15



PICTURE
OF
VALUES PLOTTED
ON
NORMAL PROBABILITY
PAPER

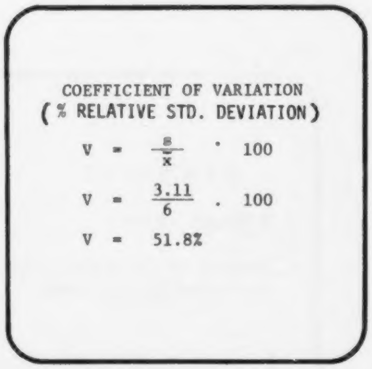
SLIDE 16



PRECISION

- 4 CONCENTRATION LEVELS
- 7 REPLICATES OF EACH
- 2 HOUR LAB TIME (min.)
  HIGH, LOW, I, I,
- CALCULATE s
- COMPARE TO METHOD s

SLIDE 17



COEFFICIENT OF VARIATION
(% RELATIVE STD. DEVIATION)

$V = \frac{s}{x} \cdot 100$

$V = \frac{3.11}{6} \cdot 100$

$V = 51.8\%$

SLIDE 18

SERIES X-24: PRECISION

RANGE
(R)

MAXIMUM VALUE
MINUS
MINIMUM VALUE

SLIDE 19

R as ESTIMATE of s

$$\frac{R}{d_n} \sim s$$

SLIDE 20 (LAST SLIDE)

SLIDE

SLIDE

SLIDE

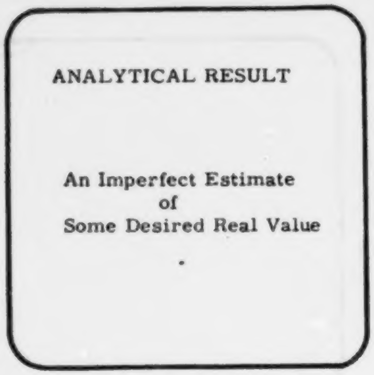
SLIDE

SLIDE

SLIDE

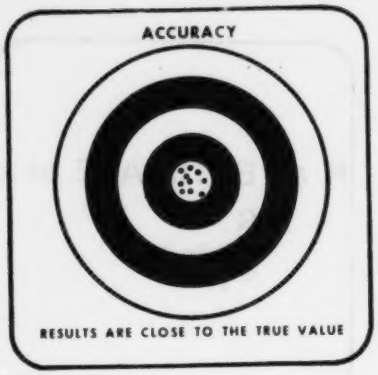
SLIDE

XI. DESCRIPTION OF VISUAL MATERIALS


ANALYTICAL RESULT

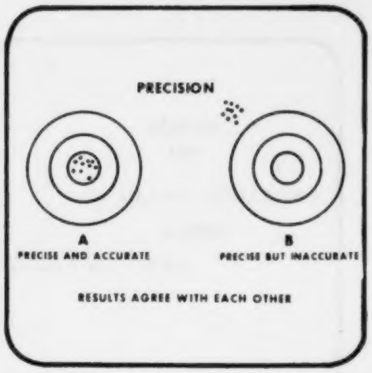
An Imperfect Estimate
of
Some Desired Real Value

SLIDE 1


ACCURACY

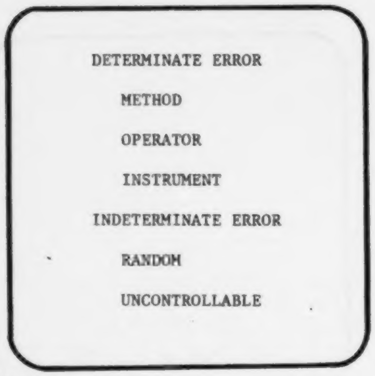
RESULTS ARE CLOSE TO THE TRUE VALUE

SLIDE 2


PRECISION

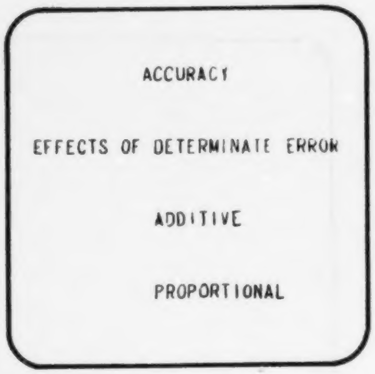
RESULTS AGREE WITH EACH OTHER

SLIDE 3


DETERMINATE ERROR
- METHOD
- OPERATOR
- INSTRUMENT

INDETERMINATE ERROR
- RANDOM
- UNCONTROLLABLE

SLIDE 4

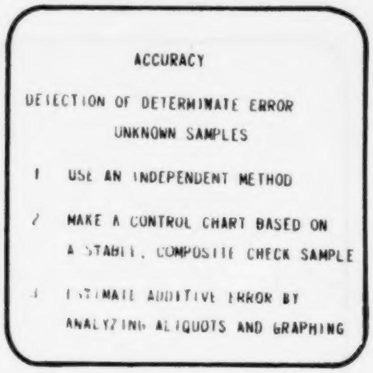

ACCURACY

EFFECTS OF DETERMINATE ERROR

ADDITIVE

PROPORTIONAL

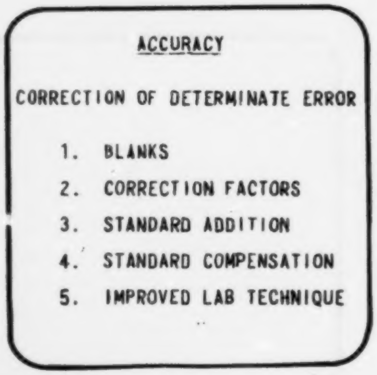
SLIDE 5


ACCURACY

DETECTION OF DETERMINATE ERROR
UNKNOWN SAMPLES

1 USE AN INDEPENDENT METHOD
2 MAKE A CONTROL CHART BASED ON A STABLE, COMPOSITE CHECK SAMPLE
3 ESTIMATE ADDITIVE ERROR BY ANALYZING ALIQUOTS AND GRAPHING

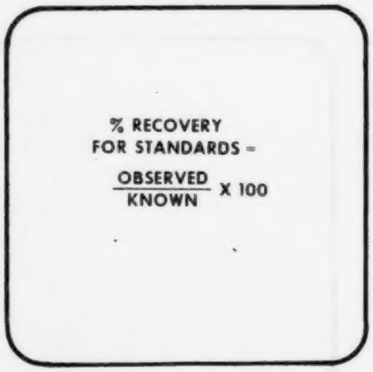
SLIDE 6


ACCURACY

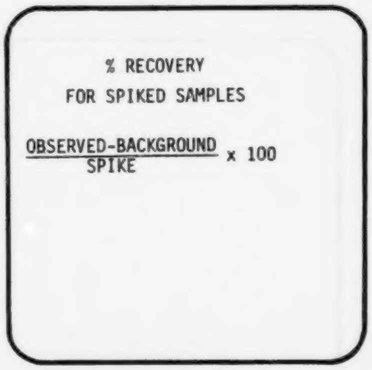
CORRECTION OF DETERMINATE ERROR

1. BLANKS
2. CORRECTION FACTORS
3. STANDARD ADDITION
4. STANDARD COMPENSATION
5. IMPROVED LAB TECHNIQUE

SLIDE 7


% RECOVERY FOR STANDARDS = $\frac{\text{OBSERVED}}{\text{KNOWN}} \times 100$

SLIDE 8


% RECOVERY
FOR SPIKED SAMPLES

$\frac{\text{OBSERVED-BACKGROUND}}{\text{SPIKE}} \times 100$

SLIDE 9

SERIES X-25: ACCURACY

"BACKGROUND"

The analytical result for the unspiked sample with regard to its volume as a constituent of the spike which yields the "observed" analytical result.

SLIDE 10

ACCURACY

- RUN SAMPLE UNSPIKED
- RUN SPIKED SAMPLE (DOUBLE LOWEST, I to 75%)
- 7 REPLICATES of EACH
- 2 HOUR LAB TIME (min.)
- CALCULATE % RECOVERY

SLIDE 11

DIFFERENT NAMES,
SAME STATISTIC:

EPA - % BIAS

ST'D METHODS
- % RELATIVE ERROR

SLIDE 12

% BIAS (% relative error)
FOR STANDARDS =

$$\frac{\text{MEAN - TRUE VALUE}}{\text{TRUE VALUE}} \times 100$$

SLIDE 13

ACCURACY STATISTICS

% BIAS *OR*
% RELATIVE ERROR
EQUALS
% RECOVERY – 100
(gives sign)

SLIDE 14 (LAST SLIDE)

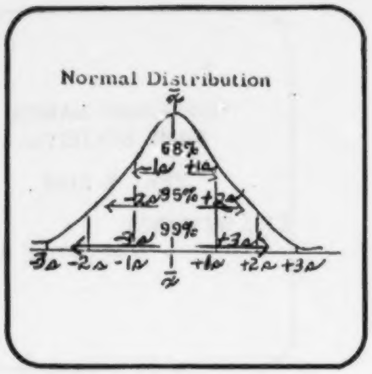
SLIDE

SLIDE

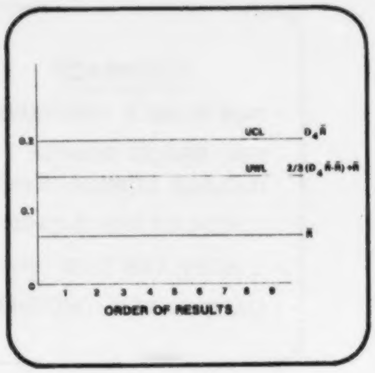
SLIDE

SLIDE

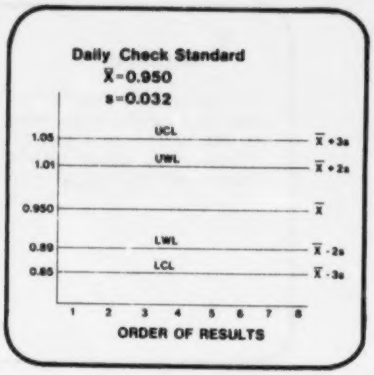
SERIES X-26: QUALITY ASSURANCE


Normal Distribution

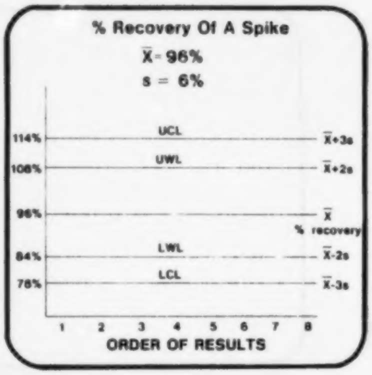
SLIDE 1



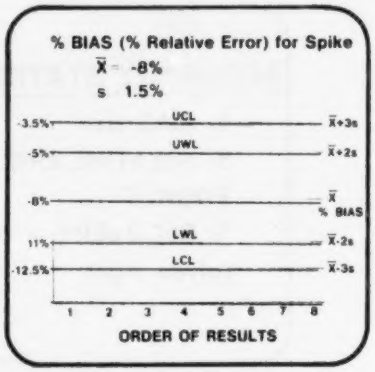
SLIDE 2


Daily Check Standard
$\bar{X}$=0.950
s=0.032

SLIDE 3


% Recovery Of A Spike
$\bar{X}$= 96%
s = 6%

SLIDE 4


% BIAS (% Relative Error) for Spike
$\bar{X}$= -8%
s 1.5%

SLIDE 5 (LAST SLIDE)

SLIDE ______

SLIDE ______

SLIDE ______

SLIDE ______

8.5* Method of Standard Additions: In this method, equal volumes of sample are added to a deionized distilled water blank and to three standards containing different known amounts of the test element. The final volumes of all the solutions must be the same. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations from the weight of analyte added to each of the original volumes of sample plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 1.

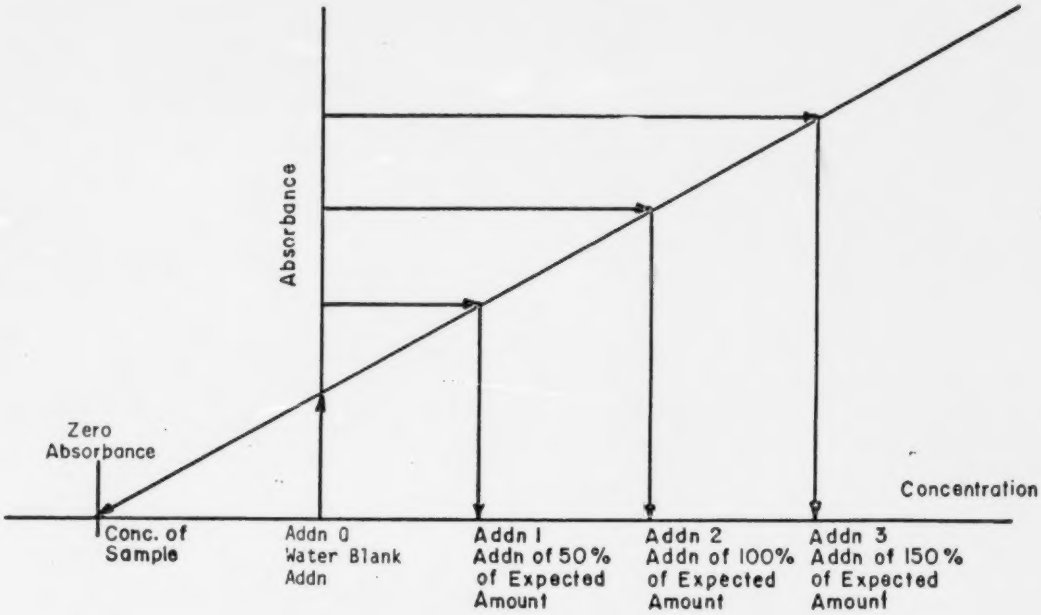


FIGURE 1. STANDARD ADDITION PLOT

The method of standard additions can be very useful, however, for the results to be valid the following limitations must be taken into consideration:

a) the absorbance plot of sample and standards must be linear over the concentration range of concern. For best results the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%) caution should be exercised.

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*from: 1979 EPA "Methods for Chemical Analysis of Water and Wastes"

b) the effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes and the standard addition should respond in a similar manner as the analyte.

c) the determination must be free of spectral interference and corrected for non-specific background interference.

EXAMPLE OF CALCULATING PERCENT RECOVERY FOR A SPIKE

A sample was collected and a 25 ml aliquot was analyzed for nitrate nitrogen. The result of the analysis was 500 µg/liter. A 25 ml spiked aliquot of the same sample was prepared using 1 ml of a spike solution which contained 10 mg/liter nitrate nitrogen. The result of analyzing the spiked sample was 898 µg/liter. Calculate the percent recovery.

$$P = \frac{\text{observed} - \text{background}}{\text{spike amount}} \times 100$$

$$\text{observed is} \left(\frac{898\ \mu g}{\text{liter}}\right) (0.025\ \text{liter}) = 22.45\ \mu g$$

$$\text{background is} \left(\frac{500\ \mu g}{\text{liter}}\right) (0.024\ \text{liter}) = 12.00\ \mu g$$

$$\text{spike amount is} \left(\frac{10{,}000\ \mu g}{\text{liter}}\right) (0.001\ \text{liter}) = 10.00\ \mu g$$

$$P = \frac{22.45\ \mu g - 12.00\ \mu g}{10.00\ \mu g} \times 100$$

$$P = 104.5\%$$

SIGNIFICANT FIGURES - EXAMPLES

32.2 has 3 significant figures

32.20 has 4 significant figures

0.0014 has 2 significant figures (zeros used to fix decimal location)

0.1400 has 4 significant figures

0.14 has 2 significant figures (the zero indicates no whole number)

*** *** *** ***

1. Retain as many significant figures in a result or in laboratory data as will give only one uncertain figure. (Possibly, in very accurate work involving lengthy computations, two uncertain figures may sometimes be retained.)

2. When rounding off, if the rejected figure is 0, 1, 2, 3, or 4, just let the remaining significant figure stand as is. If the rejected figure is 5, round the remaining figure to the nearest even number: 2.25 becomes 2.2 and 2.35 becomes 2.4. If the rejected figure is 6, 7, 8, or 9, increase the remaining significant figure by one unit.

3. As a practical operating rule for multiplication or division, keep as many significant figures in an answer as were contained in the factor having the least number of significant figures. The answer should not "suffer" if the other factors are limited to a minimum of one more significant figure than that least number of significant figures during the calculation.

4. For addition or subtraction, the number with the fewest decimal places determines the number of decimal places retained. However, no one number involved in the calculation operation limits the number of significant figures in the result.

Source: Standard Methods for the Examination of Water and Wastewater, 14th edition, APHA-AWWA-WPCF, 1975, pp. 17-20.